STUDIES ON WAXES FROM SCOTTISH SHALE OIL.

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CONTENTS

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SUMMARY.

The development of the methods used for extracting and refining waxes from Scottish shale oil is traced from the start of the industry, and details of the present method of carrying out the operations involved are given. The steps in the refining process are:- distillation of crude oil, dewaxing of waxy distillate, chemical treatment of crude scales, sweating of treated scales, decolorisation of the sweated waxes, and finally the preparation of wax cakes from the liquid product.

Each of the above operations has been investigated experimentally, and the results form the main subject of this thesis. Various grades of shale waxes have been examined in the laboratory, and the significance of certain properties from the point of view of the utilisation of the waxes is demonstrated. In each section of the work reference is made to available publications.

Crude shale oil contains 12/13% wax, and the first operation in wax manufacture is the preparation of a waxy distillate suitable for cold filter pressing. It was found that this could be obtained by excluding undesirable high boiling point constituents from the pressable distillate, and by adding wax-free oil to reduce/

reduce viscosity. Experiments showed that to prepare scales of low oil content from the charge to the filter presses, the filtration pressure should be as high as possible, and should be maintained as long as possible. Thick cakes of scale formed between buckled plates in the filter press gave higher oil contents than normal cakes. Frequent turning of buckled plates in the filter press to make them all face in one direction largely eliminated the formation of thick cakes of scale and resulted in lower oil content scales.

The extraction of wax from the pressable blend is carried out in two stages, first at 38°F. and then at 22°F. These temperatures were decided on because at 38°F. a hard scale was formed which on sweating to the highest setting point wax desired gave a product having a satisfactory oil content and "width of cut", while filtration at 22°F. resulted in a dewaxed oil of suitable setting point.

The scales extracted, amounting to 22/23% of the charge to the presses, are dark in colour and unstable, due to pyridine bases, phenols, etc. in the oil in the scale, which in the case of the hard scale amounts to 15/17%, and to about 30% in the case of the soft scale. To remove the colour, refining with sulphuric acid is applied/

applied, and experiments to determine the best conditions for this treatment proved that:-

(1) 96% sulphuric acid gave a better product than 79% sulphuric acid.

(2) The quantity of acid necessary varied with the oil in the scale, and excess was a disadvantage.

(3) The lower the temperature of treatment, the better the product.

(4) 30 minutes agitation with acid was sufficient, and long time of contact resulted in high colour.

(5) For neutralisation after acid, sodium carbonate was preferable to caustic soda.

During the sweating process oil in the scale is removed and the waxes divided into grades depending on setting point.

Sweating has been largely carried out by rule of thumb methods, and a survey of the literature on the subject shows that in no case have the results of experimental work with all relevant data been published. Since sweating is an important process in the making of waxes, this has been studied in some detail.

Because of poor separation between oil and wax in any particular sweating, a large amount of recycling must be done to obtain narrow cut waxes of low oil content.

The first essential of any sweating is that the charge should be such that when oil removal is complete/

complete the wax left will have the required setting point and "width of cut".

A scheme of recycling has been drawn up for the preparation of high quality waxes with the minimum of sweating. The basis of the scheme is that fractions should not be blended according to setting point or oil content, but according to setting point of the oil-free wax. In this way fractions containing the same grade of wax are blended for further sweating. In the preparation of shale waxes by this scheme, 4.67 tons of original charge plus recycle are sweated for every ton of finished wax produced, and the yield of finished wax is 65% of the original charge.

The plant at present in use for sweating shale waxes consists of Henderson tray sweaters and an Alanmor coil stove. It was thought that the tank type of sweater with vertical tubes might be an improvement on the existing plants. To prove this point experimental work on a semi-works scale vertical tube sweater was carried out. It was found that a vertical tube sweater 17 ft. high, and having a cooling and heating surface of 195 sq.ft. per ton of charge, could give yields and quality of products very similar to those of the Henderson House. The application of pressure above the wax being sweated was tried as a means of increasing the rate of/

of sweating, but the results were not favourable.

Detailed experiments were made to determine the optimum conditions for sweating various fractions in the experimental vertical tube sweater, and a direct comparison with the Henderson House sweater made in each case. The fractions used and the experiments made were such that conditions (rate of cooling, temperature to which charge should be cooled, rate of raising temperature during sweating, and maximum temperature to which circulating water should be raised) and yields for any fractions in the sweating scheme could be deduced.

As regards the comparison between the two sweaters, it was found that when making waxes of the same oil content, the products percentage on charge per hour (cooling plus sweating) for the tube sweater was 2.4/6.5 times that of the Henderson House, depending on the product being sweated.

The importance of accurate temperature control was noted. Time could be wasted by cooling too far and by sweating too slowly at the beginning. On the other hand, by raising the maximum temperature a degree or two above the optimum, very considerable loss in yield resulted. It was found that the only way of knowing when the proper conditions had been applied was to plot

rate of sweating (percentage on wax actually in sweater per hour - not percentage on charge per hour) against time. Under optimum conditions, the rate of sweating was fast at the start, in certain cases as high as 15% per hour, and slow towards the finish. When making single sweat products (oil content about 5%), a true rate of sweating towards the end of the process of about 4% per hour was satisfactory, but when making waxes of low oil content, a rate of about 2% per hour was found to be the maximum permissible.

The question of double sweating as against sweating in one stage was investigated, and it was proved that double sweating was the proper procedure because by this method a slightly greater yield of a slightly better wax was realised, and there was a small reduction in total time. For the minimum total time by double sweating, the first sweating should be stopped when the yield of wax is approximately 50% on charge.

The importance of maintaining the oil content of the charge to the sweating plant at a minimum was proved by tests in which scales of various oil contents were processed. Increase of oil in scale resulted in longer time of sweating, lower yield of products, and raised the setting point of the wax.

The relationship between yield of wax and

oil/

oil in charge was found to be linear, and two important points were established. The first was that at 70% oil in a charge, no wax could be separated by sweating, and the second was that the total yield of wax (that is by direct sweating and by sweating strainings completely to oil and wax) which can be realised from any charge was about 2.8 times the yield which could be obtained by direct sweating.

The sweated waxes are slightly coloured, and the final treatment applied is percolation through Floridin. The Floridin after use is washed with naphtha, steamed and ignited, and is then ready for further use. The quantity of fresh floridin added is about 2% of the Floridin actually used, and is equivalent to approximately 0.013 of a ton per ton of wax decolorised.

From tests to find the effect of certain factors on the efficiency of the process, it was shown that:-

(1) Within the limits investigated the rate of filtration had no appreciable influence on the colour of treated wax. The best colour, however, was obtained at the lowest rate, and the highest colour at the fastest rate.

(2) The lower the temperature of filtration, the better the decolorisation.

(3) Filtration of the coloured wax through Floridin which had been used but not recovered before finally decolorising was an advantage.

(4)/

(4) The best recovered Floridin was found to contain about 5% of volatile matter, and was prepared by igniting the used Floridin for 20 minutes at a temperature of 1000°F.

The casting of the liquid waxes to give cakes offered a few problems. It was found that quick cooling as in press moulding gave a wax with less mottling, but more opaque, than slow cooling, e.g. in pan moulding.

The cause of mottling in wax cakes was shown to be due to air which enters the wax during cooling and solidification, but the phenomenon was influenced by the oil content of the wax. In normal pan moulded wax excessive opaqueness and mottling were only present when the oil in the wax was high.

From a study of the published results of investigations to determine the chemical compositions of paraffin waxes in general, it appears that the normal paraffin hydrocarbons predominate with smaller percentages of the branched chain paraffin and cyclic hydrocarbons. The presence of only the former, however, has been proved in the case of wax from Scottish shale oil. During an exhaustive research made at Bristol University on shale wax of setting point 131/133°F., seven normal hydrocarbons from $C_{22}H_{46}$-$C_{31}H_{64}$ were isolated.

Various/

Various properties of different grades of wax have been determined. By observing the cooling of commercial shale waxes under specified conditions, the existence of a transition point 37°F/38°F. below the setting point was confirmed. With a narrow cut wax prepared in the laboratory by long sweating, a more definite transition effect took place at a temperature of 32.5°F. below the setting point.

The Jackson-Burmah wax testing machine, designed and developed by the staff of the Burmah Oil Co. to test the hardness of wax, and indirectly to determine oil content and "width of cut", was found very useful for checking the quality of waxes during the experiments on sweating. This instrument is described, and curves showing the effect of oil, "width of cut", and temperature on the hardness of shale waxes are given.

The significance of certain properties in relation to the utilisation of waxes is discussed.

The thesis may be summarised briefly as under:-

(a) A study has been made of the physical properties of wax, and the various factors involved in the extraction and purification of wax from Scottish shale oil.

(b)/

(b) By applying the information gained in this study many improvements have been made in existing methods of wax production. The principal development is that the industry is now installing modern vertical tube sweaters in preference to Henderson sweaters. The optimum design of such vertical tube sweaters for shale waxes has been worked out, and detailed operating conditions found for all grades of wax.

INTRODUCTION.

Historical.

The discovery of paraffin wax is generally credited to Reichenbach[1], who in 1830 isolated wax with a melting point of 110°F. from wood tar. The existence of wax in petroleum, however, had been vaguely known for some time, and in 1830 Christison[2] discovered paraffin wax in Rangoon petroleum, but Reichenbach was the first to describe the physical and chemical properties, and stated that "a wick impregnated with it burns like a fine wax candle and without smell". Reichenbach gave the material he prepared the name "Paraffin" on account of its inert nature.

In 1833 Laurent[3] experimented with bituminous shale from Autun, and this resulted in Selligue working these shales on a large scale. The products made - light oils, lamp oil, heavy oil and paraffin wax - were shown at the Paris Exhibition of 1839[4]. Selligue took out a number of patents both in France and England before 1845, covering the distillation of the shale and refining of the oil distillate. He described treatment with acid and alkali, and claimed to make white crystalline paraffin suitable for candle manufacture.

The need for a substitute for the material used at the time for candle making appears to have been widely/

widely recognised, and various attempts at preparing wax from all kinds of raw materials were made. Reece and Kane[5] in 1849 took out a patent for the manufacture of paraffin from peat, and a plant was put down at Kildare in Ireland, and later other plants were erected, but none of these worked very long.

James Young separated wax, which he made into candles, from petroleum obtained from a colliery at Alfreton in England in 1848. The supply of petroleum, however, gave out, and Young, having come to the conclusion that petroleum was produced by the effect of heat on coal, began a search for a material which on heating would give petroleum. In 1850 he patented a process[6] for "obtaining paraffine oil or an oil containing paraffine and paraffine from bituminous coals" by slow distillation. Various specimens of coal were examined, and the best results (120-130 galls/ton) were obtained from Boghead coal or Torbanhill Mineral, which was discovered near the base of the coal measures in West Lothian.

In conjunction with Meldrum and Binney, Young set up a plant in 1851 at Bathgate, West Lothian, to distil this material. The separation of wax from the oil distillate proved troublesome, and as much as possible/

possible of it was allowed to go away in the oil products, while that which separated naturally was burned. In 1854 Young started to separate the solid paraffin by artificially cooling the oil, and stored it until a practical method for dealing with it was evolved. It was not until 1859 that the wax was made into a commercial product.

About 1860 the importance of shale as a source of oil and wax had been discovered by Bell, who was granted the right to work the mineral in the Broxburn area, and subsequently shale fields were found in different parts of Scotland, but principally in the Lothians. When in 1862, owing to local consumption and the large quantity sent abroad, the supply of Torbanhill Mineral became exhausted, the Bathgate Works were supplied with shale. The oil yield from the shale then worked was about 40 gallons per ton, and to keep the refinery working at capacity, oil from other shale distilling plants was bought in.

The supply of oil shale was abundant and permitted a great expansion of the oil industry in Scotland when Young's patent expired in 1864. In that year 38 new works were built, and in 1865 no fewer than 120 works were in operation. Paraffin wax was first produced in quantity from shale oil, and for many years Scotland/

Scotland was the principal producer with a yearly make of 20,000 tons.

The industry, however, has had many ups and downs, and curtailments have frequently been necessary because of competition with petroleum, which has replaced shale oil as the chief source in the world's market of paraffin wax. At the present time the production of wax from shale oil is approximately 9,000 tons per year.

The earliest method of removing wax from the heavy distillates from the crude oil was to allow the oil to cool to atmospheric temperature in large tanks. When cold, the mass was filtered through canvas bags. This process was carried out during the winter months only and during the summer the oils were stored. The next step in the process was hydraulic pressing (200/300 lbs. per sq.in.) and treatment with sulphuric acid and soda. This method of dewaxing removed only the higher melting point waxes, and after a time the recovery of lower melting point wax by refrigeration of the filtered oil wax commenced. The oil was brought into contact with the surface of a revolving cylinder through which a cooled calcium chloride solution was pumped. The paraffin which adhered to the surface of the cylinder was continuously removed by a fixed scraper and pumped into/

into a filter press.

Various attempts were made at improving the plant for cooling the heavy oil containing paraffin. Different regrigerating agents were tried before ammonia was finally selected, and several forms of coolers were devised, notably those of McCutcheon, Henderson and Bryson. The McCutcheon cooler, introduced about 1876, consisted simply of a circular jacketed vessel fitted with a mechanically driven scraper. The oil to be cooled was pumped into the cooler and brine was circulated through the jacket.

In the Henderson cooler, patented in 1884, about 1000 gallons of oil were cooled by means of cold brine. The cooler consisted of a jacketed trough which was divided into cells by means of plates bolted together and with a space between through which the brine was circulated. After passing through these narrow spaces the brine entered the outer jacket of the vessel. The oil was kept in motion and prevented from solidifying on the surface of the cells by scrapers and by a stirrer which revolved in the curved bottom of the cooler. When the desired temperature was reached, the cooled mass was pumped to the filter presses.

The Bryson cooler was patented in 1900,

and/

and was made up of a number of concentric pipes about 20 ft. long and placed horizontally. The cooling medium (ammonia) was pumped into the annular space between the tubes, entering at the lowest, and the vapours exhausted by the compressor at the highest, while the oil containing paraffin was pumped through the inner tubes in the reverse direction continuously into the filter presses. Through the centre of the inner tube passed a shaft with scraper attached. This was revolved and removed the frozen scale from the inside of the cooler. The coolers still in use are similar in design to the Bryson cooler.

The filter presses have not altered much in principle or design until recently. The presses were the usual plate and frame type and contained from 50 to 180 plates, the maximum operating pressure being 100 lbs. per sq.in. The oil content of the scale would probably be between 50% and 60%, and to remove as much of the oil as possible, the scale was packed in strong cotton cloth and charged to hydraulic presses. Each hydraulic press contained 24 shelves on which the scale in the cloth was placed. Pressure was gradually applied to a ram under the shelves, and this forced the oil in the scale through the cloths and it dripped down into a sump underneath the press. The pressure was raised/

raised to a maximum of one ton per sq.in. and was generally maintained for about six hours. The pressure was then released, the shelves lowered, and the scale removed. The cakes formed were approximately 38" x 30" x 1" in size, and had an oil content of about 10%.

By 1868[7] recrystallisation from spirit of the crude wax from the filter presses was commenced, and this process was continued at some of the works for nearly 20 years. The crude wax or scale, as this product is called, was melted and mixed with 10/20% of spirit, and the mixture run into pans and allowed to cool. The cakes, 24" long, 12" broad and 3" thick, were wrapped in sacking and placed in a hydraulic press kept at a temperature of about 100°F. This crystallisation was performed two or three times and the wax then steamed to remove traces of spirit. The wax was finally decolorised by mechanically agitating at a temperature of about 240°F. with animal charcoal. After settling, the wax was filtered through paper to remove any char which had not settled out.

The waxes made by the repeated washings with spirit were of excellent colour and high melting point, but the process was very dangerous and frequent fires occurred. At the same time the costs were high due/

due to the unavoidably large loss of naphtha and the amount of steam required for the repeated melting of cakes and steaming of the final naphtha washed wax, with the result that sweating of wax first patented by Hodges[8] of Prices Patent Candle Co., in 1871 was largely adopted. All later developments in sweating have simply been improvements on the original idea.

The scale to be sweated was melted and run into trays to cool. When cold the cakes (about 10 lb. in weight) were placed on a cocoanut mat on a shelf in the sweating chamber, the shelves being slightly inclined in order that the sweatings would run to a collecting tank. The sweating chamber was heated by steam pipes, the temperature being kept slightly below the melting point of the scale charge; the sweating time was normally 3 to 5 days. The oil and low melting point waxes which sweated off were cast in cakes and sweated to give lower melting point wax. If the superior grade known as twice sweated wax was required, the sweated waxes were melted and resweated. The sweated product in all cases was decolorised by animal charcoal.

Because of the labour involved, this method of sweating was costly, and in 1887 Henderson[9] patented his process, which is still, with slight modifications/

modifications in some cases, used all over the world.

The sweating chamber is a brick building about 52' x 16' x 12' high with steam pipes along the walls near the bottom for heating, and large doors and ventilators at either end which can be opened for cooling. The chamber is fitted with two sets of nine superimposed horizontal trays about 21' x 6½' x 6" deep, giving the house a capacity of about 17 tons. Each tray is provided with a horizontal diaphragm of gauze about 2" from the bottom. The iron framework carrying the trays can be lowered slightly at one end to enable liquid to flow freely from the trays. The method of operation is to run water into each tray until the diaphragm is just covered, the trays being in the level position. Melted scale is next run into each tray until full, and the doors and ventilators left open until cooling is complete. This is judged by the hardness of the scale at the warmest part of the house. To commence sweating, the water in the bottom of the trays is run out, leaving the solid cake resting on the gauze. The trays are then tilted to the centre, the steam heating turned on slightly and the doors and ventilators closed. The temperature of the house is regulated to give the rate of sweating and melting point of wax desired, the strainings from all trays being collected for further sweating. When the

wax/

wax in the trays is of the quality wanted as judged by melting point of strainings leaving the sweater and by appearance, open steam is introduced into each tray under the wax. This melts the wax which runs out, and, as in the case of the previous process described to make high grade wax, two sweatings are generally necessary.

Another type of sweater was introduced by Henderson[10] in 1905. In this sweater vertical cylinders took the place of the trays, the scale being run into an annular space between two concentric tubes 9' high. The outer tube was of 17" dia. and the inner 7", and as in the case of the tray sweater a false bottom of gauze was fitted. To assist in draining during sweating, the inner tube on the side in contact with the wax was covered by a wire gauze. To begin with, the molten charge was cooled by the atmosphere, and sweating was controlled by hot air from steam pipes placed under the sweater, but later the sweaters were erected in a water bath so that cooling and sweating could be done by water circulation. Although thissweater was said to be cheaper and easier to erect, and to give a better separation of oil from the scale with resultant higher yields of finished wax, it has not been used in the shale industry during the last 15 years.

The Alanmor sweating stove, which was designed/

designed by Allan and Moore of the Burmah Oil Co.Ltd. and has been the subject of several patents since 1922,[11] has been used in refining shale waxes since 1937. This sweater, of 20 tons capacity, consists of a vertical cylindrical tank divided into 7 compartments each 30" deep and containing 14 horizontal coils of ¾" pipe at 2" pitch. Each coil is offset to the coil above and below in order to give support to the mass of wax during sweating. Through the centre of the stove passes a vertical pipe which is used for charging and removing sweatings andrunning out sweated wax. This pipe is slotted opposite the bottom of the seven compartments so that each compartment may be filled and emptied. On the floor of each compartment is placed a ¼" mesh expanded metal supported on studs. The clearance between the floor and the gauze for drainage is about ½". Liquid scale is pumped into the stove and cooling effected by pumping cold water through the coils. When the charge is cold enough, water heated to the proper temperature by means of steam is circulated through the coils, the temperature of the water being raised as sweating proceeds. The sweated product in this case cannot be seen, and sweating is therefore controlled by means of the setting point of the strainings leaving the plant./

plant. The product is run out by raising the temperature of the water high enough to melt the wax quickly. The sweater has a greater capacity than the Henderson sweater, chiefly because of the shorter time for cooling, and the finished waxes are very similar in quality and yield.

Until about 1914 the sweated wax was decolorised by the contact method already described, the decolorising agent generally used being some form of charcoal, but decolorising by percolating through ignited Fuller's Earth (Floridin) was then introduced with great success and is still in operation. After use the Floridin is washed with spirit to remove wax, steamed and reactivated by igniting in a rotary furnace. The mixture of spirit and wax is steamed, the spirit being recovered as a distillate and the wax residue is returned to process.

Since the start of the industry the first step in refining shale oil has been a distillation to coke. Young distilled the crude oil batchwise in cast iron stills of capacity about 600 gallons, but the continuous processes of Henderson, patented in 1883 and 1885, incorporating boiler stills and coking stills and the use of steam, were widely adopted and were still in use until 1936. The fractionation obtained was poor and several redistillations were/

weretherefore necessary. It was early recognised that a slight cracking of the heaviest distillate, by distilling without steam and recycling gave a solid paraffin more crystalline in structure and more easily separated during the filtration process.

Conacher[12] has given an historical account of the Scottish Shale Industry in general, while detailed descriptions of the refining of shale oil, including wax extraction by the methods in use at the time, have been given by Bailey[13] and later by Smith, Grant and Allen.[14]

Present Method of Refining Shale Waxes.

In 1937 distillation of the crude oil by means of pipe still and bubble tower was introduced. The waxy distillate, without previous chemical treatment, was dewaxed in a plant incorporating a new type offilter press. These plants and alternative methods of refining shale oil have been described by Smith and Peutherer.[15]

Wax extraction and refining are now carried out according to the scheme given in Fig.1.

Scope of Present Investigation.

Each step in the process of wax refining has been investigated and the results form the main subject of this thesis. The separation of wax cut from/

REFINING SCHEME FOR MANUFACTURE OF WAXES FROM SHALE OIL.

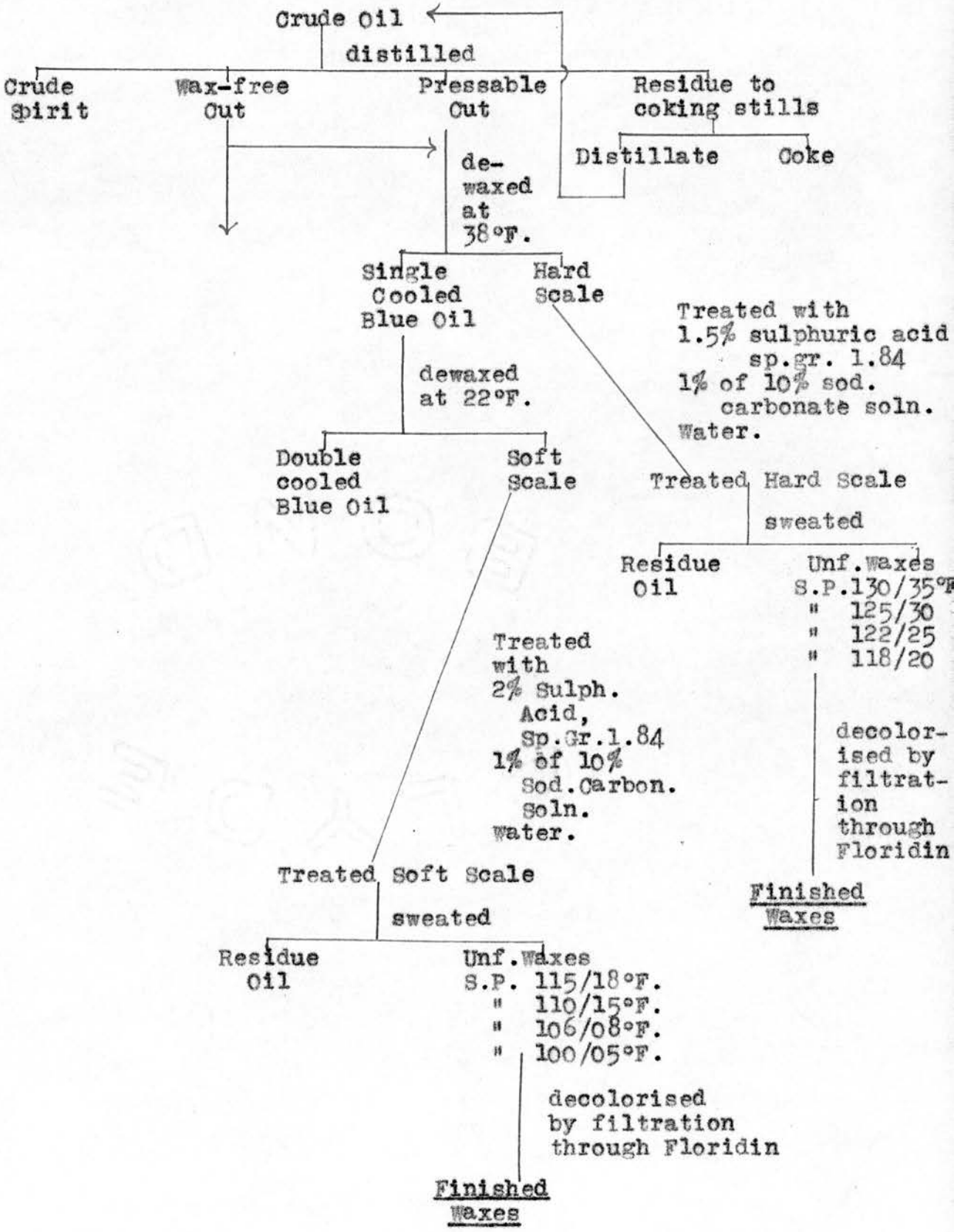


FIGURE 1

from crude oil by distillation and in particular the preparation of a distillate suitable for cold filter pressing has been studied. The conditions on the wax extraction plant to give scale of low oil content have been determined. Some experiments were made to find best conditions for the sulphuric acid treatment of scales in order to obtain a product of minimum colour.

An exhaustive enquiry was made on the problem of sweating scales to waxes of low oil content. A sweating scheme has been drawn up showing what waxes should be made, from which fractions these should be prepared and how strainings should be blended so that satisfactory waxes may result from the minimum of sweating.

Attempts have been made to improve on the technique of sweating by experimenting on a semi-works scale plant incorporating vertical cooling and heating tubes on the same lines as has been found successful by the Burmah Oil Co. The points gone into included effect of heating surface, height of sweater, application of pressure, different rates of pumping water during cooling and sweating, and finally the optimum operating conditions for sweating various fractions were determined and comparisons made with the Henderson House.

The/

The decolorisation of sweated waxes by means of Floridin has been studied.

The effect of casting liquid waxes under different conditions on the appearance of the cake of finished wax has been investigated with particular reference to the cause and prevention of "mottling".

In the section dealing with chemical and physical properties of shale waxes, the literature on the chemistry is reviewed and analyses (setting point, transition point, specific gravity, distillation range, flash point, aniline point, oil content and hardness number) of various grades are given.

The use of and interpretation of results from the Jackson-Burmah wax testing machine are described. This instrument was devised by the research staff of the Burmah Oil Co. to test hardness of waxes and indirectly to give a measure of oil content and "width of cut". Curves showing effect on hardness of oil, temperature, blending ofwaxes of different setting point and addition of Stearine are given. Throughout this section the significance of the constants determined from the point of view of the utilisation of the waxes is demonstrated.

WAX EXTRACTION.

(1) Preparation of Pressable Cut.

Crude shale oil contains 12/13% wax, and the first step in extraction of the wax is the preparation of a waxy distillate suitable for cold filter pressing.

Distillation of Crude Oil.

Crude oil was distilled in the laboratory under good fractionation conditions at pressures ranging from 15 mm. mercury to normal to give a pressable cut and an amorphous wax fraction. This second fraction was made suitable for adding to the pressable cut by redistilling under mild cracking conditions. The total waxy distillate was then dewaxed by filtration at low temperature (20°F) and it was found that the lower the pressure at which the crude was distilled the greater the yield of wax. The yields of oil free wax at the different pressures were as under:-

TABLE 1

	Distillation Pressure		
	15 mms. mercury	330 mms. mercury	Atmospheric
Oil-free Wax, % on crude oil,	13.8	11.9	10.2

Distillation of the crude oil in the refinery has been described by Smith & Peutherer[15], and consists briefly of distillation in a pipe still and bubble tower/

tower in presence of steam to a 15% residue, which is then taken to coke in pot stills. Amorphous wax which distils in the last 10% of the crude is concentrated in the 15% residue. By controlling the conditions under which this residue is coked, sufficient cracking is induced to convert the amorphous constituents into crystalline paraffin, and the distillate is added to crude oil as recycle.

Crystallisation of Wax Distillate

The necessity for good fractionation between pressable and amorphous wax has long been recognised and is in line with experience in petroleum refining. Much published work is available on the crystallisation of waxy distillates. A review of the work of various authors is given by Carpenter[16], while recent contributions to the knowledge of the subject are due to Bruter[17] and to Padgett[18], the latter being one of the first to point out the importance of good fractionation and elimination of entrainment.

The presence of small quantities of amorphous wax or asphaltic matter in a waxy distillate results in the formation of small crystals with subsequent poor filtration and high oil content scales. Too high a viscosity or too high a concentration of wax in the distillate also hinder crystallisation, and these are remedied/

remedied by the addition of the requisite quantity of wax-free oil. The addition of wax-free oil, however, to a badly fractionated pressable cut will not result in any improvement in pressing quality. Experience in the refining of shale waxes is in agreement with the conclusions of Padgett that when the pressable distillate is of suitable quality the difficulties in all subsequent operations disappear.

Methods available for controlling the quality of the charge to filter presses are - by pressing on the laboratory scale, by distillation at a pressure of 40 mm. mercury, and by observing crystallisation under the microscope. A convenient method for carrying out the microscopic examination is given in detail by Davis and Campbell.[19]

The normal pressable cut from crude shale oil has a viscosity of 80 seconds at 100°F. and contains about 27% of wax. This fraction when dewaxed at 38°F. gave a scale having an oil content of 24%, and by adding wax-free oil to the pressable cut before dewaxing in the ratio of 1:2, a scale containing only 17% oil was obtained. The addition of this wax-free oil reduced the wax in the mixture to about 18% and lowered the viscosity to 50 seconds at 100°F. The effect on the dewaxed oil of adding this wax-free oil was to reduce the viscosity at 70°F/

70°F. from 260 seconds to 70 seconds, and at 40°F. from 790 seconds to 150 seconds.

Analyses of Pressable Cut.

By controlling conditions on the crude oil distillation plant to give a well fractionated press cut, and adding wax-free oil as above, a blend having good pressing qualities is obtained. Analyses of a satisfactory pressable distillate and of the corresponding blend are given below:-

TABLE 2

	Pressable distillate	Blend of:- Press.Dist. / Wax-free Oil	2 parts / 1 part
Sp.Gr. at 60°F.,	.894	.880	
Setting Point, °F.	90	80	
Viscosity (Redwood 1) at 100°F., secs.	80	50	
Distillation Range at 40 mms. Hg			
I.B.P.	124°C.	99°C.	
10% distillate at,	225 "	155 "	
20% " "	239 "	194 "	
30% " "	248 "	220 "	
40% " "	256 "	237 "	
50% " "	265 "	251 "	
60% " "	275 "	265 "	
70% " "	284 "	279 "	
80% " "	296 "	293 "	
90% " "	310 "	310 "	
F.B.P.	325 "	323 "	
Total distillate,	98.0%	98.0%	
Residue,	1.0%	1.0%	
Loss,	1.0%	1.0%	

The results are more or less in agreement with experience in the American Petroleum industry.

Davis/

Davis and Campbell[19], for example, examined pressable distillates, before being diluted with wax-free oil, from various Mid Continent refineries, and found the Final Boiling Point at 40 mm. mercury to range from 333°C. to 342°C. The same authors fractionated at 3 mm. mercury an Oklahoma crude oil which had previously been topped to remove all wax-free oil. The wax distillate was collected in fractions and it was found that a distillate of Final Boiling Point 344°C. (at 40 mm. mercury) gave a good crystal structure, but by increasing the Final Boiling Point to 358°C. the distillate showed distinct evidence of poor crystalline form.

(2) Dewaxing of Pressable Distillate.

In this operation as conducted at present the blend of pressable cut and wax-free oil is cooled and dewaxed in two stages, the scales obtained being refined separately. The first filtration takes place at a temperature of 38°F. and the filtered oil is then cooled to 22°F. and again filtered. The first filtration is carried out at 38°F. because it was found that at this temperature the scale separated gave, on sweating to the highest setting point wax desired, a wax of satisfactory oil content and "width of cut"*. The temperature of 22°F. for the second filtration gave a/

* range of setting points

a dewaxed oil which, when blended with wax-free oils and redistilled, resulted in the finished oils having the necessary setting point and cloud point. If the temperature of the second cooling is allowed to fall two or three degrees below that given, a considerable increase in oil content of the scale is noticed, and filtration rates become slow.

The pressable blend is well settled to remove water and suspended matter. The former, if present, would form ice in the coolers which might damage the mechanism, while the latter would tend to clog up the filter cloths.

In all early references to wax refining the importance of slow cooling to allow the wax crystals time to grow was emphasised, but the same importance is not now placed on this point. Padgett[18], in his recent publication, states that the rate of chilling cannot be discussed positively and that many refiners find no advantage by slow cooling.

Description of Coolers and Filters.

The plants for dewaxing at each stage consist of a heat exchanger, two coolers and two filters, with the necessary pumps and ammonia compressors. In the heat exchangers the oil is cooled by means of cold filtered oil, and in the coolers the cooling medium is liquid/

liquid ammonia. These coolers are somewhat similar to the Bryson coolers already described, but are capable of standing much higher pressures, and the cooler is kept full of liquid ammonia by means of a constant level device. The gaseous ammonia produced during the cooling is drawn off by a compressor, compressed, cooled and returned to the cooler.

The filter presses are of the horizontal hydraulic plate type with loose rings. There are 510 plates of 3'9" dia. in each press, and rings ½" thick are placed between each plate. The plates have a solid steel centre plate 3/16" thick having projecting lugs to support the plate in the press. There is a hole in the centre 6" dia. On either side of the plate is placed a light perforated steel plate which also has a hole at the centre. The perforated plates are covered by the filter cloth and all parts of the filter plate are held together by means of 12 rivets and 24 buttons. The object of the perforated plates is to keep the cloth away from the solid centre plate and so obtain free drainage of the oil which has passed through the cloth. The buttons on all plates are placed in the same position so that they are directly opposite each other when the plates are in the press. The plates and rings are brought tightly together by means of a hydraulic ram and/

and are then held by tie rods and nuts.

The chilled mass enters the filter at one end and passes through the centre holes, filling the spaces between the plates. The filtered oil runs into a trough under the press, and when scale is being removed from the filter the trough is removed mechanically and its place is taken by a conveyor to take away the scale.

Filter presses of this type are now widely used throughout the oil industry, and a very detailed description (including diagram and photographs) and data on operation, together with a discussion on the merits and disadvantages, are given by Espach[20], who states that the oil content of scale made in America varies from 30% to over 60%.

The oil to be dewaxed is pumped through the heat exchanger into a second pump which forces the oil through the cooler and into the filter. The pressure at the filter inlet rises to a maximum of 450 lbs./sq.in. This gives a pressure of 600 lbs./sq.in. on the cooler, which is the maximum allowable working pressure for the plant.

When the rate of flow of filtered oil is very slow, i.e. when the scale space in the filter is full, the press is opened up and the scale between the rings is dropped into the conveyor beneath the plates. The/

The average rate of filtration including time taken for emptying the filter works out at 0.128 gl. per sq.ft. of filtering area per hour. In the second stage filtration, i.e. when cooling to 22°F., it is found that the rate of filtration gradually becomes slower, and the scale sticky and oily, due to the pores of the cloths clogging up. To overcome this, the filter cloths are cleaned regularly, about once a month, by pumping hot oil through the filter. A flow diagram of the wax extraction plant is given. (Fig.2).

Oil Content of Scales.

Under normal conditions the first stage scale (hard scale) has a setting point of 118°F. and expressible oil content of 15/17%, while the scale (soft scale) from the second cooling and pressing has a setting point of 97°F. and expressible oil content of about 30%. The total scale extracted amounts to 22/23% of the oil charged to the plant, the ratio of first scale to second scale being 2:1. The twice cooled blue oil has a specific gravity of .890 and a setting point of 20°F.

The effect of operating conditions on the oil content of scales was determined experimentally. With a maximum pressure on the filter of 350 lbs./sq.in. the oil in the scale amounted to 20%, and by increasing the pressure/

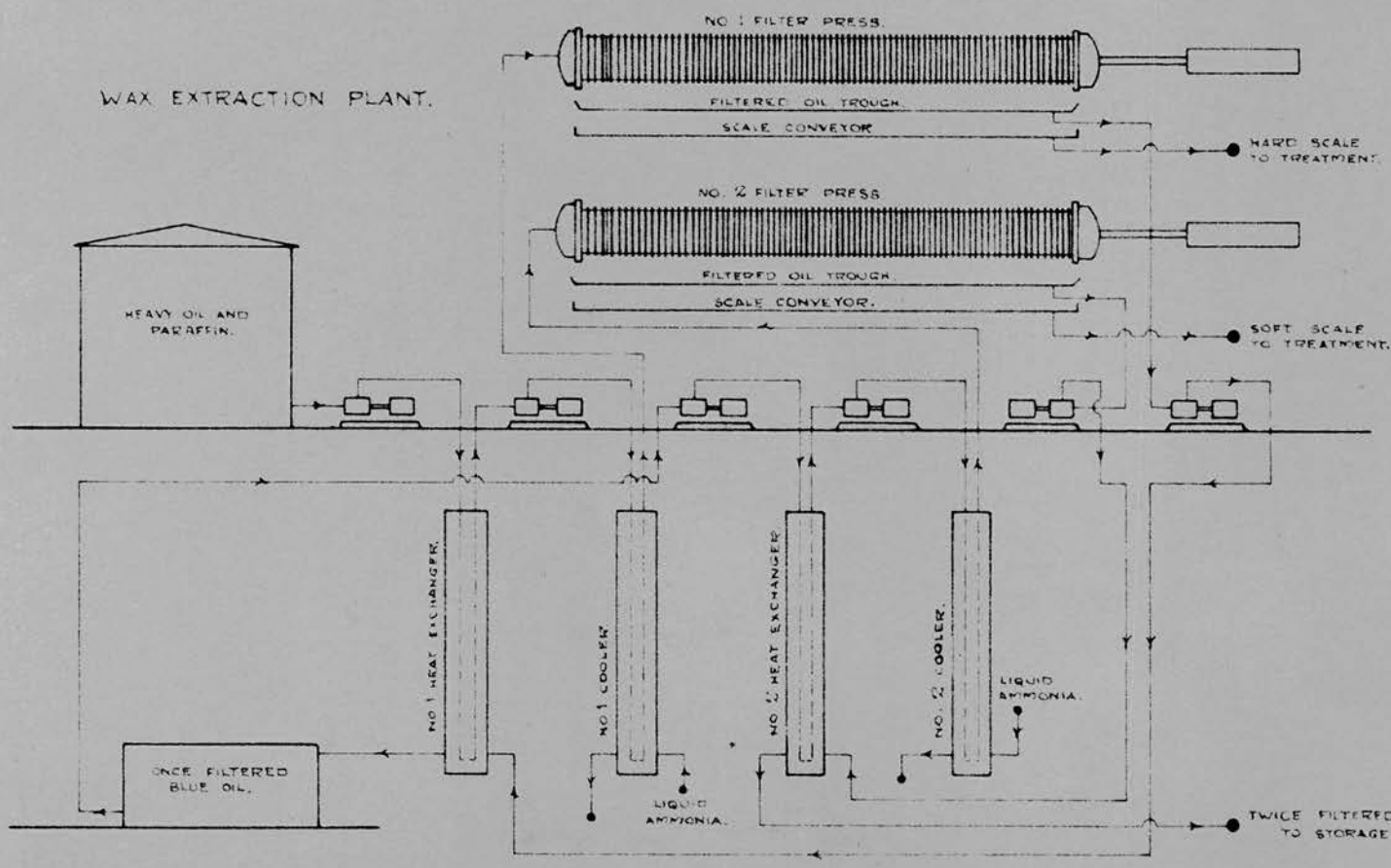

WAX EXTRACTION PLANT.
NO. 1 FILTER PRESS.
FILTERED OIL TROUGH.
SCALE CONVEYOR.
HARD SCALE TO TREATMENT.
NO. 2 FILTER PRESS.
FILTERED OIL TROUGH.
SCALE CONVEYOR.
SOFT SCALE TO TREATMENT.
HEAVY OIL AND PARAFFIN.
NO. 1 HEAT EXCHANGER.
NO. 1 COOLER.
NO. 2 HEAT EXCHANGER.
NO. 2 COOLER.
LIQUID AMMONIA.
LIQUID AMMONIA.
ONCE FILTERED BLUE OIL.
TWICE FILTERED OIL TO STORAGE.

FIG.2

pressure to 400 lbs. per sq.in., the oil in the scale was reduced to 17%. The time for the total cycle of operations on the plant was 12 hours. The time for draining and removing scale was 3 hours, leaving 9 hours for pumping. By pumping the oil into the filter at such a rate that it took 6 hours to reach a maximum pressure of 400 lbs.per sq.in. and then maintaining the pressure for 3 hours, the scale had an oil content of 21.5%, whereas by raising the pressure to a maximum of 400 lbs. in 3 hours and maintaining this pressure for 6 hours, the oil in the scale amounted to 17.8%. To raise the pressure to the maximum in 3 hours it was necessary to work the pumps faster than they were designed for, and so the next test was to raise the pressure at such a rate that a maximum of 450 lbs. was reached in 4 hours. The maximum pressure was maintained for 5 hours in this case, and the scale obtained had an oil content of 16.5%.

A few tests were made using an experimental hydraulic press to find the effect on the scale of time, pressure and thickness of cake. When a pressure of 1120 lbs./sq.in. was maintained for 8 hours, the oil in the scale amounted to 14.9%, and by doubling the time under pressure, the oil content of the scale was reduced to 13.6%. By increasing the pressure to 2240 lbs/sq.in. and keeping the pressure on for 8 hours, a scale/

scale with an oil content of 11% was obtained. The quantity of scale charged to the press was reduced to give a cake half the normal thickness, and for comparison cakes of normal thickness were also prepared. The thinner cake was found to contain 7.0% oil, while the normal cake contained 8.8%.

As a result of all the tests it is concluded that to obtain a scale of minimum oil content, (and that ought to be the function of the wax extraction plant) it is necessary that the pressure be as high as possible and maintained for as long as possible, and that thick cakes of scale should be avoided.

Plate Buckling.

The question of thick and thin cakes is important, because one of the drawbacks to this type of filter press is buckling of the plates, and this leads to thick and thin cakes. Thick cakes of scale when tested were found to contain 33% oil, while normal sized cakes from the same filtration showed only 13%. Frequently plates become so badly bent that thick cakes and empty spaces occur alternately in a filter press. A considerable amount of experimental work has been done in an attempt to overcome this trouble. It is considered that the buckling is due to a higher pressure being formed on one side of the plate than on the other, and if for any reason/

reason some plates filtered more readily than others unequal pressure would result. It was found that buckling could be reduced by making several cuts through the centre solid plate at its edge to a depth of 2" to 3", that is far enough to extend inside the joint made by the ring when the press is built up. These slots result in free drainage from the plate, and at the same time give equal pressures on both sides. One way of overcoming the trouble of the thick and thin cakes is to turn the buckled plates so that they all face in one direction. In this way, although the plates are buckled, normal sized cakes result. Unfortunately this is not a permanent cure, but it has been found to be satisfactory to turn the plates requiring to be turned once a week. The time taken to do this is about two hours.

CHEMICAL TREATMENT OF CRUDE SCALES

The hard and soft scales from the filter presses are very dark in colour and unstable. This is due to the pyridine bases, phenols and unsaturated hydrocarbons present in the oil in the scale. To remove these impurities treatment with sulphuric acid is necessary, and experiments have been made to find the optimum conditions for this treatment. In the refinery, the treatment is carried out in vertical cylindrical vessels and agitation is by air blowing.

Determination of Optimum Conditions of Treatment

Hard scale was treated with (1) 1.5% by vol. sulphuric acid, sp.gr. 1.84 (96%), and (2) 2.0% by vol. sulphuric acid, sp.gr. 1.72 (79%), under the same conditions as regards temperature, time of treatment and settling. The acid treated scales were neutralised by shaking with excess 10% sodium carbonate solution. The colours of the treated scales ($\frac{1}{2}$" cell) were 5.5Y+0.3R for treatment (1) and 20.0Y+2.0R for treatment (2). The treated scales were sweated to give unfinished waxes of setting point 125°F. under exactly similar conditions, the colours of the waxes (6" cell) being (1) 10.0Y+1.2R and (2) 23.0Y+2.0R.

Treatment with 1.5% sulphuric acid of sp.gr. 1.84 /

1.84 is very similar as regards quantity of pure sulphuric acid to treatment with 2.0% sulphuric acid of sp.gr. 1.72, and the smaller quantity of the stronger acid was found to give a much better coloured treated scale than the larger quantity of the weaker acid. On sweating, the lower coloured scale gave the better wax.

In the subsequent tests, therefore, only acid of sp.gr. 1.84 was used, and the factors investigated were (1) quantity of acid, (2) time of acid treatment, (3) temperature of acid treatment, (4) time of settling after acid, and (5) neutralisation after acid with sodium carbonate as against caustic soda. The tests in each section were made under strictly comparative conditions, and the results are summarised in Table 3.

The results showed that 2.0% sulphuric acid (sp.gr. 1.84) gave the lowest colour, and treatment should be carried out for 30 minutes. The temperature should be as low as possible - about 135°F. The acid tar settled quickly and so neutralisation should take place after about one hour settling. Long settling on acid should be avoided. Sodium carbonate after acid gave a lower colour than caustic soda, and excess sodium carbonate did not affect the colour. Sodium carbonate solution also tended to give less stable emulsions than caustic/

TABLE 3

TREATMENT OF CRUDE SCALES UNDER DIFFERENT CONDITIONS

Quantity of Sulph.Acid (Sp.Gr.1.84) used	Temperature of treatment	Time of treatment	Time of settling after acid	Quantity of 10% Sodium Carbonate soln.used	Quantity of 10% Caustic Soda soln.used	Colour of treated scale after filtering through paper ($\frac{1}{2}$" cell)
Quantity of Sulphuric Acid.						
1.0%	135°F.	20 mins.	30 mins.	1.5%	-	10.0Y+1.5R
1.5%	"	"	"	"	-	6.2Y+0.6R
2.0%	"	"	"	"	-	4.2Y+0.3R
2.5%	"	"	"	3.0%	-	4.0Y+0.4R
3.5%	"	"	"	"	-	4.2Y+0.45R
Time of Acid Treatment.						
2.0%	135°F.	10 mins.	30 mins.	1.5%	-	4.0Y+0.3R
2.0%	"	30 "	"	"	-	3.5Y+0.3R
2.0%	"	60 "	"	"	-	4.0Y+0.35R
Temperature of Acid Treatment.						
2.0%	135°F.	30 mins.	30 mins.	1.5%	-	3.5Y+0.3R
2.0%	160°F.	"	"	"	-	5.0Y+0.6R
2.0%	210°F.	"	"	"	-	5.5Y+0.8R
Time of Settling after Acid.						
2.0%	135°F.	30 mins.	30 mins.	1.5%	-	3.5Y+0.3R
2.0%	"	"	1½ hours	"	-	3.5Y+0.3R
2.0%	"	"	18 "	"	-	18.0Y+4.0R
Neutralisation of Acid Treated Scale.						
2.0%	135°F.	30 mins.	30 mins.	1.5%	-	3.5Y+0.3R
2.0%	"	"	"	-	1.5%	6.5Y+1.0R
2.0%	"	"	"	10.0%	-	3.5Y+0.3R

caustic soda. To completely remove sodium carbonate, two or three washing with hot water were necessary. This water washing was found to be important because scales containing alkali gave trouble in the sweating process. The scale on cooling did not give a hard cake, but was softer than usual, and oil removal was difficult.

The oil content of scale treated under the above conditions came down from 17.5% to 15.0%.

The effect of storage on the colour of treated scale was also investigated. A quantity of scale was treated under best conditions and stored for 14 days at a temperature of 130°F. The colour of the scale was determined at intervals, and these were as under:-

TABLE 4

	Colour of treated Scale ($\frac{1}{2}$" cell)
When produced,	3.5Y+0.3R
After 24 hrs. @ 130°F.	4.0Y+0.3R
" 7 days " "	5.0Y+0.5R
" 14 " " "	5.5Y+0.6R

This would indicate that storage time between treatment and sweating should be as short as possible.

Tests made with soft scale gave very similar results, but on account of the higher oil content generally about 0.5% more acid was required than for hard scale.

SWEATING OF TREATED SCALES

The work on sweating of treated scales, herein described, is divided into four sections, (1) sweating scheme, (2) experiments in connection with design of a vertical tube sweater, (3) determination of optimum conditions for sweating various fractions in the vertical tube sweater and comparison with Henderson House, and (4) general conclusions on sweating.

(1) Sweating Scheme.

The treated scales contain, as already mentioned, 15/30% oil, and this must be removed and the waxes divided into grades depending on setting points. This is achieved at the sweating plant. During sweating liquid scale is charged to the sweater, where it is cooled to a definite point, and heat then gradually applied. Sweating is really fractional fusion, and so the first fraction to melt and be removed consists of oil with low setting point wax dissolved in it. The proportion of wax to oil in the strainings gradually increases, and the setting point of the wax in the oil also increases (see Table 5). The period of sweating must be such that the wax left in the sweater at the finish of the process is practically free from oil - generally less than 1.0%.

During the sweating a considerable quantity of wax is melted out. This is as important as the oil removal since the finished wax should not, except where specially desired, consist of a long range of setting point waxes./

waxes. For this reason the starting material for any sweating should be such that when the oil removal is completed a wax is left having the necessary setting point and range of setting point.

TABLE 5

Oil Content, Setting Point, and Setting Point Oil-free of Strainings leaving Sweater at hourly intervals.

The charge, hard scale, had a setting point of 119.7°F. and oil content 15.9%, while the wax left in the sweater (single sweat wax) after 12 hours sweating showed setting point 126.5°F. and oil content 6.5%.

	Oil Content %	Setting Point °F.	Setting Pt. Oil-free °F.
After 1 hour,	56.8	89	103
" 2 hours,	56.6	90	104
" 3 "	60.6	92	107
" 4 "	58.1	94	109
" 5 "	48.9	96	108
" 6 "	43.7	98	109
" 7 "	41.0	100	110
" 8 "	35.6	102	111
" 9 "	33.2	104	112
" 10 "	30.2	106	113
" 11 "	27.2	107	114
" 12 "	24.4	108	114

Because of the quantity of wax removed during the sweating along with the oil as strainings, a large amount of recycling must be done to obtain the maximum yield of wax. Each charge is divided into strainings and wax, and if strainings leaving the sweater at the start of the process are high in oil (about 70%), these are not recycled but sent to cracking stock. The cut out setting point for this fraction, termed residue oil, in all cases is 85°F., and fractions having setting points above/

above 85°F. are recycled. When the yield of wax from any sweating would be very low before the wax is of the desired oil content, the sweating is stopped, the wax melted out, charged to another sweater and again sweated. This procedure is termed double sweating.

From the point of view of making narrow cut waxes of low oil content for the minimum amount of sweating, it is essential that the scheme of recycling be arranged so that all fractions containing the same grade of wax (that is fractions having the same oil free setting point) be blended, and that the wax to be made from each fraction be such that the sweating time will be adequate to give the necessary quality (oil content and "width of cut") when the setting point of the wax is reached.

From the literature on sweating, it appears that the scheme of blending and recycling has been given very little consideration. Espach[20], after giving a few schemes used in America, sums up by saying that each refinery has an individual way of disposing of and finishing the products from scales, and states that at some plants, where the intermediate cuts are sweated and re-sweated to highly refined waxes, the process seems almost endless.

It is sometimes suggested that 1st and 2nd scales be blended before sweating[21]. In most cases this must/

must be the wrong procedure, because a wax of satisfactory oil content from such a mixture would not have the necessary narrow cut, and to obtain this the low setting point constituents would have to be sweated off. Suppose a hard scale of oil content 15% and setting point 118°F. be sweated to give a wax containing about 0.5% oil, the setting point of this wax would be approximately 132°F. By adding soft scale of same oil content as the hard scale, a mixture having a setting point of 110°F. and 15% oil could be prepared. By sweating to a setting point of 124°F. a wax of oil content about 0.5% would be obtained, but it is obvious that this wax would be a much longer cut wax than the wax made by the first method. In addition to this definite disadvantage, there does not appear to be anything to be gained by blending the two scales before sweating.

The method sometimes used is to blend fractions roughly according to setting point. It can be shown that this is wrong by considering what would happen in the case of blending two fractions of setting point say 120°F., one having an oil content of 5% and the other 50%. The former would contain wax of setting point 121°F.(calculated) and the latter 132°F. (calculated), and so the temperatures required for sweating would be different. Another point is that the duration of sweating would require to be such that the high oil content fraction gave a satisfactory wax. Before this would be accomplished the fraction containing oil-free/

oil-free wax of setting point 121°F. would be sweated off and therefore this fraction would have been sweated needlessly.

If blending is done on the basis of oil content very similar objections hold, and the proper method is to blend fractions containing oil-free wax of similar setting points. The scheme given in Fig.3 has been worked out for shale waxes on this basis.

When the unfinished wax is decolorised by Floridin about 20% of the wax charged is left in the filters. This wax is dissolved in naphtha and the naphtha steamed off. The wax so obtained requires a further sweat, chiefly to remove the smell of naphtha. Since 20% is a very large proportion of the wax produced, it is important to return this wax to the proper place in the sweating scheme. For this reason the returns are put into a fraction which will ensure that the returned wax is sweated to a higher setting point, and again they are really blended with fractions which contain the same setting point wax.

Analyses of the scales and various straining fractions referred to in the sweating scheme (Fig.3) are given below (Table 6). These analyses form the basis on which the sweating scheme was built up.

TABLE 6/

SWEATING SCHEME FOR SHALE WAXES.

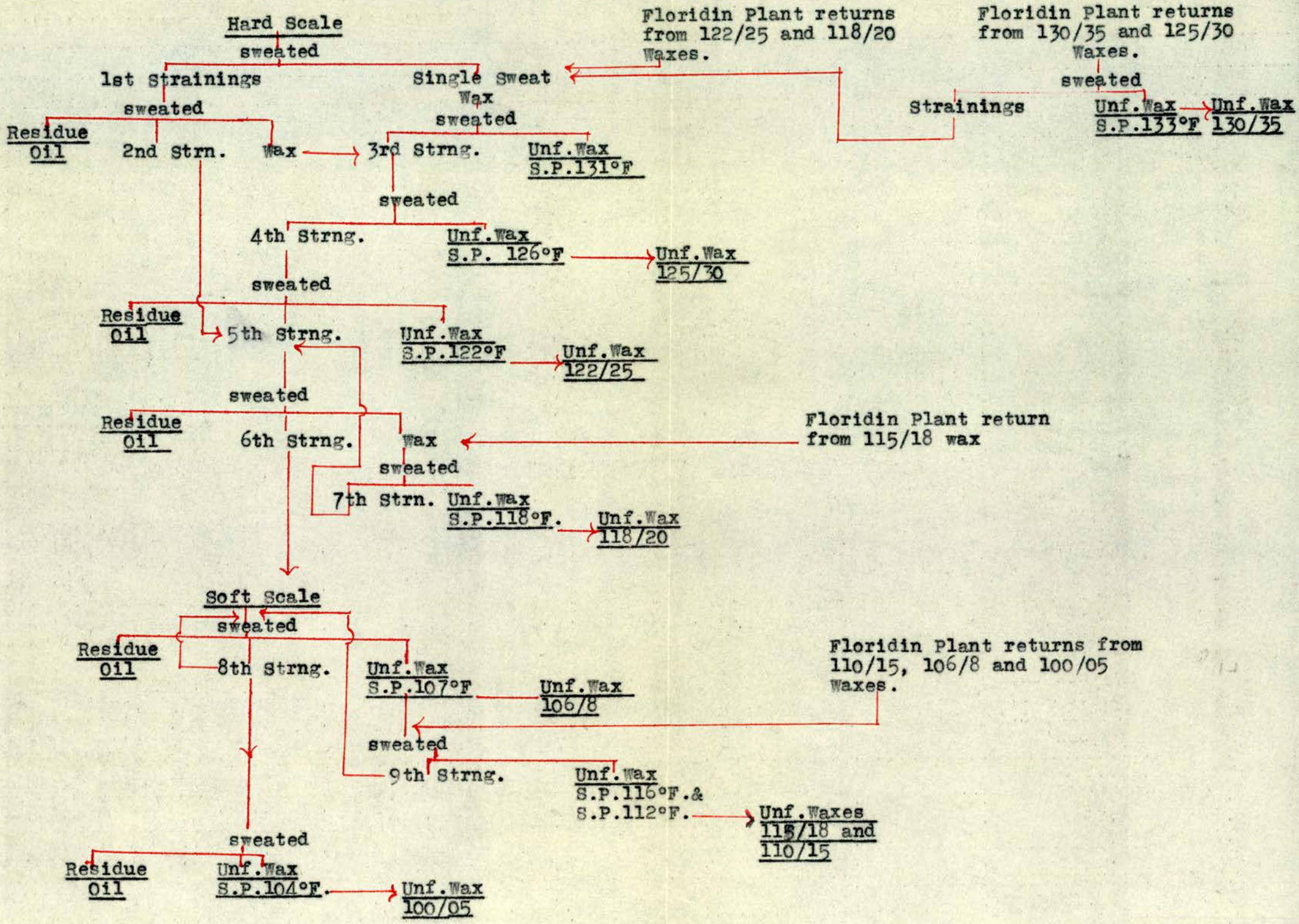


FIGURE 3.

TABLE 6

Analyses of Scales and Strainings.

	Sett.Pt. °F.	Oil Content % by wt.	Sett.Pt. of Oil-free Scale °F.
Hard Scale,	118	15	122
Single Sw.Wax from hard scale,	126	5	127
1st Strainings,	102	40	112
2nd Strainings,	98	44	109
Single Sw.Wax from 1st Strainings,	121	6	123
3rd Strainings,	121	7	123
4th Strainings,	116	14	119
5th Strainings,	102	24	108
Single Sw.Wax from 5th Strainings,	113	12	116
6th Strainings,	92	48	104
7th Strainings,	107	17	111
Soft Scale,	97	30	104
8th Strainings,	90	35	99
Single Sw.Wax from soft scale,	107	2	107
9th Strainings,	101	7	103
Residue Oil,	78	72	96

Table 7, based on average figures over a long period, showing the quantity of each fraction charged to the sweaters expressed as tons per ton of finished wax, gives some idea of the amount of sweating carried out and of relative quantities of the various fractions.

For every ton of finished wax produced 4.67 tons of material have to be sweated. The original charge to the sweating plant (hard scale + soft scale) amounts to 1.54 tons per ton of wax, which is equivalent to a wax yield of 65%. The remainder - 35% of original charge/

charge - is residue oil.

TABLE 7.

	Tons/ton of Finished Wax.
Hard Scale,	1.02
1st Strainings,	0.31
Single Sw.Wax from hard scale,	0.82
Floridin plant returns from 130/35 and 125/30 waxes,	0.10
3rd Strainings,	0.56
4th Strainings,	0.28
5th Strainings,	0.23
Single Sw.Wax from 5th Strngs.,	0.12
Soft Scale (0.52) including 6th, 8th and 9th Strainings,	0.80
Single Sweat Wax from soft scale,	0.40
8th Strainings,	0.03
Total:	4.67

Espach[20], in his survey, gives figures varying from 8.37 tons of material sweated per ton of wax to 3.5 tons per ton, but the figures cannot be related to one anothermor to the figure of 4.67 tons per ton given above, since the amount of sweating will depend largely on the oil content of the charge and on the quality of the finished waxes. Espach says that an analysis from time to time of the amount of sweating done at any plant will give a figure that measures its efficiency. This is true only if the refining scheme is not changed, as in tests which are described later it will be seen that frequently the most efficient method of operation is to sweat fast and recycle large quantities of strainings. In experiments comparing single sweating and double sweating it is demonstrated that the better method/

method in every respect for certain cuts is the double sweating. This again is a case of the most efficient method giving higher quantities to be sweated.

Products of Sweating.

The products of sweating (unfinished waxes) range in setting point from 130/35°F. to 100/05°F. The quantities of the different grades calculated as a percentage on the original charge to the sweating plant and as a percentage of the total wax made are given in Table 8, while analyses of different waxes are given in the section dealing with chemical and physical properties.

TABLE 8.

Grade of Wax. Setting Point °F.	% on original charge to sweating plant	% on total wax produced.
130/35	5.9	9.1
125/30	27.0	41.5
122/25	11.8	18.2
118/20	5.1	7.8
115/18	4.6	7.1
110/15	5.5	8.4
106/08	4.2	6.5
100/05	0.9	1.4
	65.0	100.0

The other product of the sweating plant is the oil (residue Oil) which is removed from the scales. This amounts to 35% of the original charge and has on an average a setting point of 78°F. and oil content of 72%.

(2) /

(2) Experiments in connection with Design of a Vertical Tube Sweater.

The plant at present in use for sweating shale waxes consists of 24-18 ton Henderson Houses and one 20 ton Alanmor stove, descriptions of which have already been given (pages 22-25). In operating the Henderson House type of sweater, several disadvantages have become apparent, the chief being the long time necessary for cooling and the uncertainty of cooling, since this depends on atmospheric conditions. It is necessary to cool the charge until the part furthest from the doors is sufficiently hard, and by the time this is accomplished the bulk of the charge is much colder than is required. This is very evident in cold and stormy weather, and results in time being lost during the sweating period. The heating of the chambers is also irregular. The top trays always sweat faster than those at the bottom, with the result that by the time the bottom trays are properly sweated there is a very poor yield in the top trays. A considerable amount of dust from the atmosphere collects in the pans and restricts the flow of sweatings. For this reason each tray must be frequently examined during the sweating, and this requires a fair amount of attention.

None of the disadvantages possessed by the Henderson House apply to the Alanmor stove, since cooling is by water circulation and the sweater is a closed/

closed vessel.

The yields and quality of products from the two sweaters are very much alike, while the steam used for sweating and melting out the sweated wax in the case of the Alanmor stove is only about one third of that for the Henderson House.

Much experimental work has been done with a view to designing new sweaters. The ideal plant would be a continuous sweater, but while such plants of novel design have been patented[22,23] none have ever reached the stage of commercial operation, and so the continuous process could not be considered as an alternative to existing plants. To overcome the difficulty of cleaning spiral coils, it was decided to experiment with a sweater fitted with vertical straight tubes which could be easily cleaned and removed if necessary. Sweaters fitted with vertical tubes have been in use in America for some time, and are described by Espach.[20] These are termed "tank sweaters" and normally are about 13' high, with the diameter varying from 12' to 50'. The tubes are generally 1" or 1¼" diameter and are placed at 4" to 6" centres. The sweaters are fitted with horizontal wax supports placed 4" to 6" apart. These supports are perforated plates, and the wax is thus essentially broken up into a number of 4" or 6" cakes. These plants are/

are said to give as good results as the Henderson House type but are mostly used for sweating of oily fractions or scales. Vertical tube sweaters have recently been introduced by the Burmah Oil Co.

To determine such points as cooling and heating surface required, effect of height of sweater, sweating under increased pressure and effect of pumping water at different rates during cooling and sweating, two experimental plants were erected and tests made.

(a) First Experimental Sweater. (Heating surface 77 sq.ft./ton)

Description of Plant.

The stove consisted of a rectangular vessel 4'9" x 5' x 5' high, capable of holding 2½ tons of scale. Water was introduced into the bottom to a depth of 3", and at this point was attached a perforated plate on which the cooled mass rested when the water had been removed.

Cooling and sweating were effected by pumping water through vertical tubes ¾" i.d., placed with their centres 4" apart. This gave a cooling and heating surface of 77 sq.ft. per ton of charge. The water entered at the bottom and left at the top.

The scale to be sweated was pumped into the sweater at the top, a vent being provided to allow the displaced air to escape. The strainings passed through the perforated plate and ran out at the bottom.

Steam for melting out the sweated product was introduced into the wax space at the bottom, and the melted/

melted wax was run out the same connection as was used for strainings. The outlet from the sweater was connected to four vessels of suitable size to collect the products of sweating. To eliminate the effect of atmospheric conditions the sweater was lagged.

Method of Operation.

Water was pumped into the bottom up to the level of the perforated plate, the quantity being fixed by means of an overflow. The sweater was filled with scale to be sweated and cooling commenced by pumping cold water through the tubes. When the charge had been cooled to the desired temperature the water bottom was run off and sweating started by circulating warm water in a closed system through the tubes. The temperature of this circulating water was controlled by injecting steam. When the wax in the sweater was at the desired setting point as shown by the setting point of the strainings leaving, the circulating water was shut off and steam introduced into the sweater to melt out the sweated product.

For control purposes records were taken every half hour during cooling and sweating of temperature of water entering and leaving, and the temperature of wax near the top and near the bottom. The setting point of the strainings leaving the sweater was also determined every half hour during the sweating period, while analyses of charge and of the products were carried out every run.

In/

In all experiments on sweating described in this thesis, several runs were made for eadh test and the results quoted are the average of the results obtained. In some cases preliminary runs were necessary to determine certain operating conditions. The results of such tests are not given nor are they included in the average results.

Throughout the tests on sweating, the terms used for charge and products are the same as those given in the sweating scheme (Fig.3), and by referring to the scheme it should be possible to see to what stage in the scheme the particular experiments refer.

Results.

Hard scale was sweated at three different rates to single sweat wax, and for comparison a similar charge was sweated under normal conditions in a Henderson House. Details of these tests are given in Table 9.

The experimental sweater gave yields of single sweat wax varying from 66.4% on charge to 72.1%, the waxes having oil contents varying from 4.0% to 6.6%, the poorer the wax the higher the yield. The cooling time was about 13 hours, and sweating varied from 28½ hours to 9½ hours. The Henderson House on the same charge took 30 hours to cool and 28 hours sweating, and gave a single sweat wax of oil content 4.7% amounting to 69.9% on charge. In Tests Nos.2 and 4 the waxes are of similar quality, and so a comparison between the two sweaters can be made. The experimental/

experimental sweater took 25¼ hours (cooling and sweating) against 58 hours for the Henderson House, the yields of wax being 68.6% and 69.9% respectively.

It will be noticed that in Test No.2 the cut out setting point was 112°F. to give a wax of setting point 124.3°F., while in the Henderson House test a cut out setting point of 108°F. gave a wax having a setting point of 126.5°F. This may be accounted for by the high maximum temperature reached in the circulating water during sweating.

By referring to Fig.6 it can be seen that there was a considerable difference between water temperature and wax temperature during sweating, and this would indicate that the heating surface was insufficient. This point was further demonstrated by the fact that when changing from cooling water to warm water for sweating, two to three hours elapsed before the wax temperature began to show any increase.

From the tests made it appeared that while the experimental sweater gave results not quite so good as the Henderson House, improvements could be expected by considerably increasing the cooling and heating surface.

(b) Second Experimental Sweater. (Heating surface 195 sq.ft./ton)

Description of Plant.

Since it was thought from the tests made that increased heating surface would be an advantage, a second/

second sweater was built in which the cooling and heating tubes ($\frac{3}{4}$" inside dia.) were placed with their centres 2.7/8" apart instead of 4" as in the first plant. This gave a heating surface of 195 sq.ft. per ton of charge.

The first sweater was 5' high, and it seemed desirable to find what effect there would be on the sweating of making the sweater very much higher. At the same time it was thought that the introduction of compressed air to the top of the wax being sweated might reduce the time, and the new plant was made suitable for checking these points.

The sweater consisted of a cylindrical vessel 2' in diameter and 17' high, with a capacity of one ton. In other respects the plant was similar to the one already described, and the method of operation was exactly the same.

Details of the construction of this sweater are given in Fig.4, while Fig.5 shows the complete plant.

(c) Effect of Cooling and Heating Surface.

Hard scale of the same quality as used in earlier experiments (Tests 1-3 in first experimental sweater) was sweated to single sweat wax in the second sweater, and for convenience the results (Test No.5) are given in Table 9 along with the results for the previous tests. The sweater was filled to capacity and no compressed air was applied.

The/

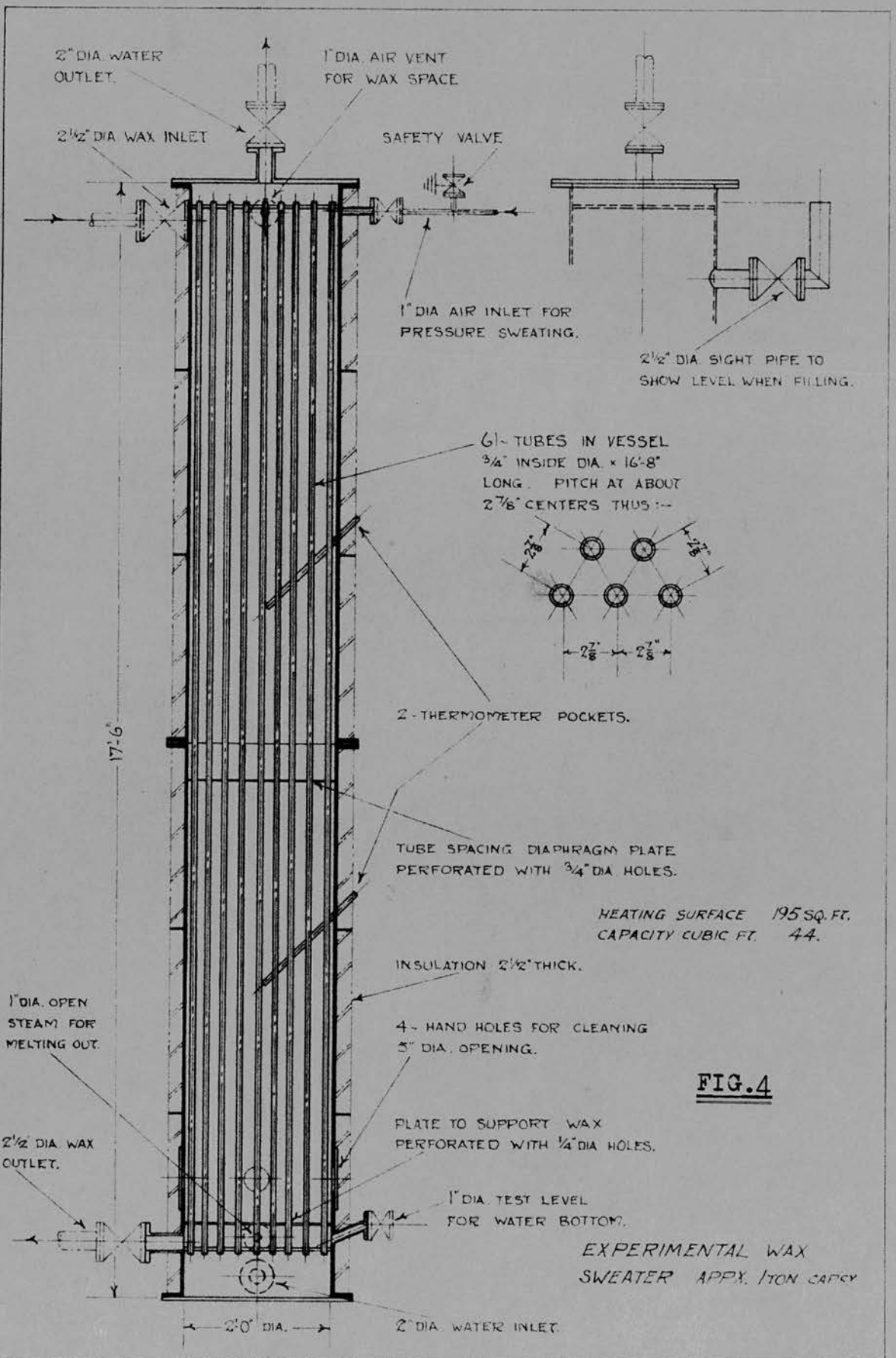

2" DIA WATER OUTLET.
1" DIA AIR VENT FOR WAX SPACE
2½" DIA WAX INLET
SAFETY VALVE
1" DIA AIR INLET FOR PRESSURE SWEATING.
2½" DIA SIGHT PIPE TO SHOW LEVEL WHEN FILLING.
61 - TUBES IN VESSEL ¾" INSIDE DIA. × 16'-8" LONG. PITCH AT ABOUT 2⅞" CENTERS THUS :-
2⅞"
2⅞"
2⅞"
2⅞"
2⅞"
2 - THERMOMETER POCKETS.
17'-6"
TUBE SPACING DIAPHRAGM PLATE PERFORATED WITH ¾" DIA HOLES.
HEATING SURFACE 195 SQ. FT.
CAPACITY CUBIC FT. 44.
INSULATION 2½" THICK.
1" DIA. OPEN STEAM FOR MELTING OUT.
4 - HAND HOLES FOR CLEANING 5" DIA. OPENING.
FIG.4
PLATE TO SUPPORT WAX PERFORATED WITH ¼" DIA HOLES.
2½" DIA. WAX OUTLET.
1" DIA. TEST LEVEL FOR WATER BOTTOM.
EXPERIMENTAL WAX SWEATER APPX. 1 TON CAPCY
2'-0" DIA.
2" DIA WATER INLET.

FIG. 5

TABLE 9

SWEATING HARD SCALE IN EXPERIMENTAL SWEATERS AND IN HENDERSON HOUSE

	1st Exptl. Sweat.	1st Exptl. Sweat.	1st Exptl. Sweat.	Henderson House	2nd Exptl Sweat
Test No:	1	2	3	4	5
Charge:	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale
Gallons,	732	720	720	5749	288
Temperature, °F.	148	148	150	140	143
Setting Pt., °F.	118.4	118.4	118.4	119	119
Oil content, %	13.0	13.0	13.0	13.3	13.
Times:					
Cooling, hours,	12½	13	12½	30	3
Sweating, "	28¼	12¼	9¼	28	11
Total "	40¾	25¼	21¼	58	14
Yields: % on charge,					
1st Strainings,	33.6	31.4	27.9	30.1	30.7
Single sweat Wax,	66.4	68.6	72.1	69.9	69.3
Conditions:					
Temp.to which charge cooled, °F.	85	85	85	-	85
Temp.of water at start of sweat. °F.	90	100	100	-	90
Temp.of house at start of sweat. °F.	-	-	-	88	-
Max.temp.of water during sweating °F.	118	125	132	-	112
Max.temp.of house during sweating, °F.	-	-	-	119	-
Rate of increase in temp., °F/hour,	1	2	3.5	-	2 till max.
Setting Pt.at cut out,	112	112	112	108	108
Rate of Sweating. % on chg./hour,	1.2	2.6	4.1	1.1	3.1
Analyses of Products:					
1st Strainings.					
Setting Pt., °F.	100	101	102	98	100
Oil content, %	42	46	41	45	43
Single Sweat Wax.					
Setting Pt., °F.	125.1	124.3	123.7	126.5	125.9
Oil content, %	4.0	5.1	6.6	4.7	4.8

The conditions were similar to those for Test No.2, and the single sweat wax amounted to 69.3% of the charge and had an oil content of 4.8%. In Test No.2 the yield was 68.6% and the wax contained 5.1% of oil. The provision of increased cooling and heating surface reduced the cooling time to 3 hours and the sweating time to 11 hours. The maximum temperature of circulating water was only 112°F. while the cut out setting point was the same as that for the Henderson House - 108°F. When changing from cooling water to warm water for sweating there was no lag as in the case of the previous sweater.

Fig.6 shows graphically the rate of sweating, temperature of circulating water and temperature of wax against time for Tests 1, 2, 3 and 5. By comparing Test No.2 with No.5 it will be seen that the increased heating surface has decreased very considerably the difference in temperature between circulating water and wax.

The slightest change in temperature of circulating water immediately showed in wax temperature, and the setting point of strainings leaving the plant at any time was the same as the temperature of the wax.

From the operation of the plant it was decided that the heating surface - 195 sq.ft./ton of charge - that is $\frac{3}{4}$" tubes with centres 2.7/8" apart, was satisfactory.

In all subsequent tests the second

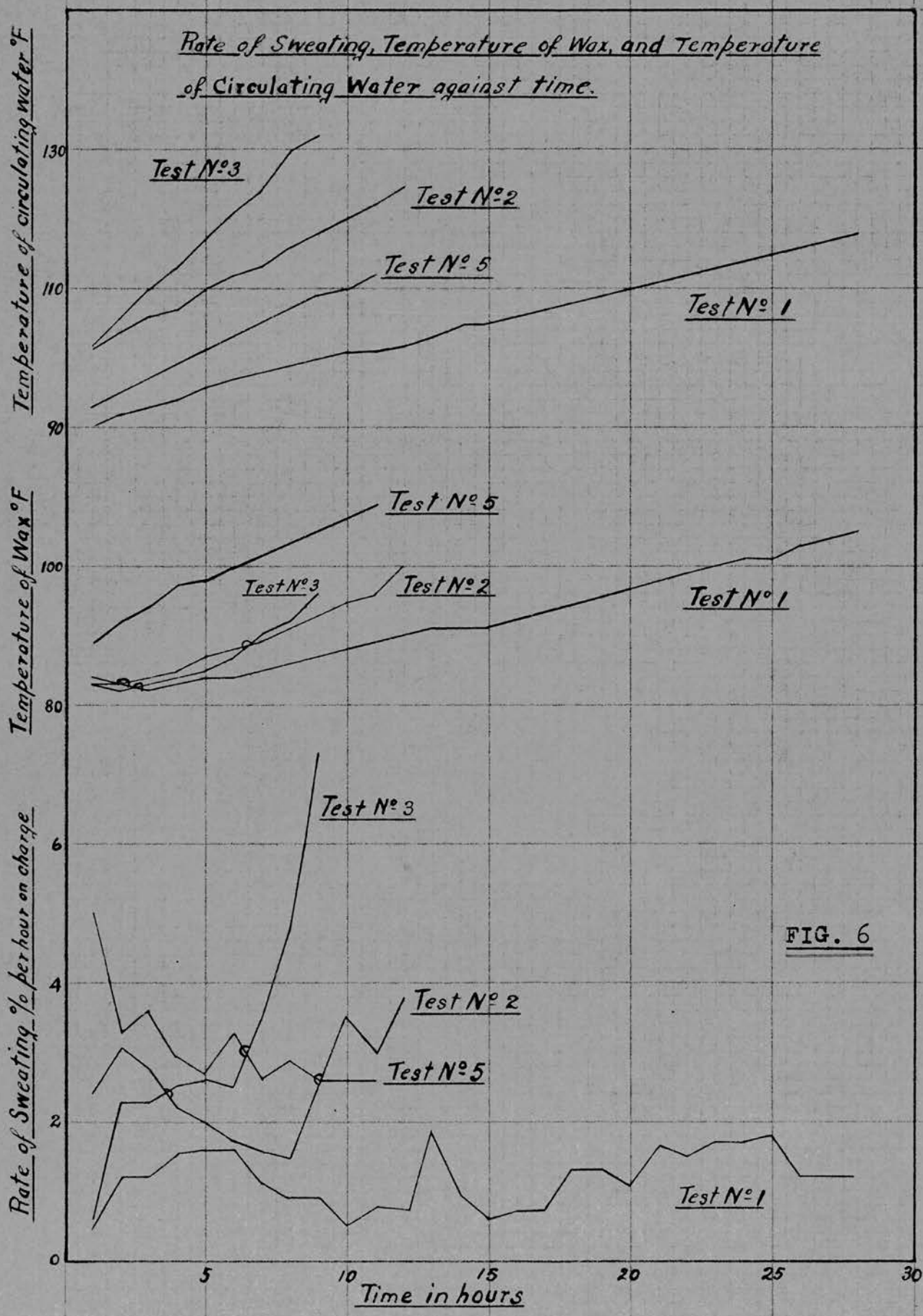

Rate of Sweating, Temperature of Wax, and Temperature of Circulating Water against time.
Temperature of circulating water °F
130
110
90
Test No. 3
Test No. 2
Test No. 5
Test No. 1
Temperature of Wax °F
100
80
Test No. 5
Test No. 3
Test No. 2
Test No. 1
Rate of Sweating % per hour on charge
6
4
2
0
Test No. 3
Test No. 2
Test No. 5
Test No. 1
5
10
15
20
25
30
Time in hours

FIG. 6

experimental sweater was used.

(d) Effect of Height of Sweater.

In the Henderson Houses the maximum depth of charge is 6", while the Alanmor stove is divided into cells, each 2'10" high. The American "tank sweaters" already mentioned are 13' high, but the wax is divided into sections each 4" - 6" deep. The first experimental sweater, which in spite of the poor cooling and heating surface gave results reasonably near those of the Henderson House was 5' high, and had no wax supports. The second sweater was made 17' high to find if a column of wax of this height could be satisfactorily dealt with.

To find the effect of height of wax column, comparative tests were made in which the sweater was filled to maximum capacity (Test No.6) and only half charged (Test No.7). Hard scale, setting point 118°F. and oil content 22.3%, was sweated to single sweat wax in each case, and the results are given in Table 10.

When the sweater was filled to capacity the single sweat wax amounted to 64.6% on charge and had a setting point of 127.3°F. and oil content 4.6%. With the sweater containing only half a charge, the yield was 63.9% and the product had a setting point of 126.7°F. and oil content 5.4%. The rate of sweating in the first case was 3.4% per hour and in the second case 3.3% per hour. These results show that there was no disadvantage, from the point of/

of view of yield and quality of product, in operating a sweater containing a column of charge about 17' high.

The comparison already given in Table 9 shows that the 17' sweater gave yields and quality of products very similar to the Henderson House.

(e) Effect of Sweating under Increased Pressure.

The same hard scale as was used in Tests 6 and 7 was sweated to single sweat wax with 5 lbs. per sq.in. and 10 lbs. per sq.in. air pressure applied.

The charge was cooled in the normal way and when sweating was started compressed air was admitted to the sweater above the charge. A safety valve, adjusted to maintain 5 lbs. and 10 lbs. pressure as required during the sweating period, was fitted,

The results of Tests Nos.8 and 9 are given in Table 10 and can be compared with Test No.6 which was under normal conditions.

The application of increased pressure above the wax did reduce the time of sweating. Under normal conditions the sweating time was 10½ hours, whereas under 5 lbs. pressure the time was 9 hours, and under 10 lbs. pressure 8 hours. The corresponding rates of sweating were 3.4%, 3.6% and 4.5% per hour.

Under normal conditions the single sweat wax amounted to 64.6% on charge and contained 4.6% oil. When sweating under 5 lbs. added pressure the yield was 67.6%/

TABLE 10

SWEATING HARD SCALE TO SINGLE SWEAT WAX - (1) NORMAL CONDITIONS, SWEATER FILLED TO CAPACITY, (2) SWEATER HALF CHARGED, (3) 5 lbs./sq.in.PRESSURE APPLIED, and (4) 10 lbs/sq.in.AIR PRESSURE.

Test No.	6	7	8	9
Conditions of Sweating.	Normal: filled to capacity	Sweater half charged	5 lbs/ sq.in. air pressure	10 lbs/ sq.in. air pressure
Charge.	Hard Scale	Hard Scale	Hard Scale	Hard Scale
Gallons,	280	150	285	287
Temperature, °F.	163	158	143	153
Setting Point, °F.	118	118	118	118
Oil content, %	22.3	22.3	22.3	22.3
Times:				
Cooling, hours,	4½	2½	4¼	3½
Sweating, "	10½	11	9	8
Total "	15	13½	13¼	11½
Yield: % on charge,				
1st Strainings,	35.4	36.1	32.4	36.3
Single Sweat Wax,	64.6	63.9	67.6	63.7
Conditions:				
Temp.to which charge cooled, °F.	85	85	85	85
Temp.of water at start of sweating, °F.	90	90	90	90
Max.temp.of water during sweating, °F.	112	112	107	106
Rate of increase in temp. °F/hour,	2	2	2	2
Setting Point at cut out, °F.	108	106	108.4	113
Rate of Sweating. % on charge/hour	3.4	3.3	3.6	4.5
Analyses of Products:				
1st Strainings.				
Setting Point, °F.	97.2	100.6	101.4	102.9
Oil content, %	56.0	45.8	48.9	45.6
Single Sweat Wax.				
Setting Point, °F.	127.3	126.7	125.2	125.5
Oil content, %	4.6	5.4	6.6	7.2

67.6%, but the oil in the product was 6.6%. When 10 lbs. pressure was applied the yield was only 63.7% and the oil in the wax 7.2%.

These figures would point to poorer products being made as a result of the increased pressure. It will be observed that it was necessary when pressure was applied to run to higher cut out setting points. Under normal conditions a cut out setting point of 108°F. gave a wax of setting point 127.3°F. When 5 lbs. pressure was introduced a cut out of 108.4°F. gave a wax having a setting point of 125.2°F., and when 10 lbs. pressure was applied a cut out of 113°F. resulted in a wax of only 125.5°F. setting point. The only explanation of these facts is that wax must have gone off in the strainings, and it was concluded that the application of pressure was not to be recommended.

(f) Effect of Rate of Pumping cold water on cooling time and on the products of sweating.

A few experiments were made to find what was the effect on the time required for cooling of pumping cold water at different rates through the tubes. At the same time the opportunity was taken to find if the rate of cooling had any effect on the subsequent sweating.

The times required to cool hard scale to 85°F. when pumping various quantities of cooling water are given in Table 11.

TABLE 11/

TABLE 11

Test No.	Rate of pumping cooling water, gls/hr/ton chg.	Temp. of charge to sweater, °F.	Temperature of cooling water		Temp. of charge after cooling °F	Time taken to cool hrs.
			Entering sweater °F	Av. temp. of water leaving °F		
10	3180	153	38	53	85	2¼
11	1780	146	37	51	85	2¼
12	890	154	38	55	85	2½
13	440	133	40	60	85	3
14	220	151	40	79	85	3¾
15	110	146	38	85	85	4¼

In order to find what effect, if any, cooling at different rates would have on the sweating, the hard scales cooled at the rates given above (Table 11) were sweated under usual conditions to give single sweat waxes. Hard scale was also cooled gradually over a period of 19 hours and then sweated under the same conditions as in the other tests. The details of these tests are given in Table 12, and from the yields and analyses of the waxes, it is evident that there is no advantage in slow cooling. To obtain maximum throughput for any plant, therefore, cooling should be effected as quickly as possible.

(g) Effect of Rate of Pumping Warm Water during Sweating.

In order to check up the effect on sweating time of pumping various quantities of circulating water during sweating, tests were made in which hard scale was sweated to single sweat wax, all conditions being the same except that the warm water was pumped at different rates.

The/

TABLE 12

EFFECT ON SWEATING OF HARD SCALE OF COOLING AT DIFFERENT RATES.

Test No:	10	11	12	13	14	15	16
Rate of pumping cooling water, gls/hr/ton scale,	3180	1780	890	440	220	110	-
Time taken to cool, hours,	2¼	2¼	2½	3	3¾	4¼	19
Charge:	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale
Gallons,	271	272	273	277	273	265	265
Temperature, °F.	153	146	154	133	151	146	151
Setting Point, °F.	119.7	119.7	119.7	119.7	119.7	119.7	119.7
Oil content, %	15.9	15.9	15.9	15.9	15.9	15.9	15.9
Times:							
Cooling, hours,	2¼	2¼	2½	3	3¾	4¼	19
Sweating, "	11	11¼	11	12¼	11¼	11¾	12¼
Total "	13¼	13½	13½	15¼	15	16	31¼
Yields of Products: % on charge.							
1st Strainings,	31.4	31.2	29.3	32.0	28.3	29.7	30.3
Single Sweat Wax,	68.6	68.8	70.7	68.0	71.7	70.3	69.7
Conditions:							
Temp.to which charge cooled, °F	85	85	85	85	85	85	85
Temp.of water at start of sweating, °F.	90	90	90	90	90	90	90
Max.temp.of water during sweating, °F.	112	112	113	114	113	114	114
Rate of increase in temperature, °F/hour,	2	2	2	2	2	2	2
Setting Pt.at cut out, °F.	108	108	108	108	108	108	108
Rate of Sweating: % per hour on charge,	2.8	2.6	2.6	2.6	2.7	2.7	2.5
Analyses of Products:							
1st Strainings.							
Setting Point, °F.	100	98	99	99	99	101	101
Oil content, %	55.4	52.2	52.0	49.2	47.3	49.1	41.6
Single Sweat Wax.							
Setting Point, °F.	126.5	126.2	126.2	126.2	125.6	125.4	125.2
Oil content, %	8.5	8.5	8.2	8.2	10.0	7.3	7.4

The wax yields and oil contents showed no difference throughout the series, and the essential points from the tests are given in Table 13.

TABLE 13

	Test No.17	Test No.18	Test No.19
Rate of pumping water during sweating, gls/hr/ton of charge,	2960	220	110
Time taken to sweat, hrs.	11¼	14¼	15¼
Rate of sweating, %/hour on charge,	2.6	1.9	1.9
Max.temp.of water during sweating, °F.	112	118	120

The greater the quantity of water pumped the shorter the sweating time, and this in spite of the fact that the maximum temperature of the water was highest at the lowest rate of flow. These results are what would be expected, that is, for minimum time of sweating and minimum temperature of water, the quantity of water circulated should be as high as possible. It is realised that in a full scale commercial plant there is a limit to the quantity of water which it would be feasible to pump, but the fact remains that the quantity should be as great as possible.

(3) Determination of Optimum Conditions for sweating various Fractions in the Vertical Tube Sweater and Comparison with the Henderson House.

Sufficient work has been described to show that the vertical tube sweater, 17' high and having a cooling and heating surface of 195 sq.ft. per ton of charge, could/

could sweat hard scale to single sweat wax, and without having determined the optimum conditions the yields and quality of products appeared very similar to those of the Henderson House, and the time cycle considerably less. Tests were next made to determine the best operating conditions for this sweater when dealing with different charges, a comparison in each case being made with the Henderson House.

The fractions used and the experiments made were such that conditions (temperature to which charge should be cooled, rate of raising temperature during sweating and maximum temperature to which circulating water should be raised) and yields for any fraction in the sweating scheme could be deduced. Hard scale was sweated to single sweat wax, which contains about 5% oil, and high setting point (131°F) and low oil content waxes were prepared from single sweat wax. The oiliest fraction dealt with at the sweating plant, 1st Strainings, was sweated to a product containing about 5% oil to find if the tube sweater would give good separation of low setting point residue oil. Fifth strainings were sweated to wax of setting point about 118°F. and low oil content, and finally soft scale, which contains a fairly high percentage of oil, was processed to give low setting point (107°F), low oil content wax. In the case of 5th strainings and soft

scale/

scale, residue oil was also a product.

In addition, a comparison of double sweating and single sweating was made and an investigation carried out to find the effect of oil content of hard scale on the products and the times necessary for sweating.

The tests are described under the following headings:-

(a) Sweating hard scale to single sweat wax.
(b) Sweating single sweat wax to wax of oil content 0.5%.
(c) Comparison of double sweating and single sweating of hard scale.
(d) Sweating hard scales of various oil contents.
(e) Sweating 1st strainings.
(f) Sweating 5th strainings.
(g) Sweating soft scale.

In all tests described in this section the quantity of cooling water pumped was 1780 gallons per hour per ton of charge, and the circulating water was 2960 gallons per hour per ton.

(a) Sweating Hard Scale to Single Sweat Wax.

Hard scale, setting point 120°F. and oil content 18.6%, was sweated at various rates and conditions to give single sweat wax of setting point about 126°F. and oil content 6/7%.

Rate of raising temperature of circulating water.

In the first series of tests (Nos.20-24) the charge was cooled to 85°F. and sweating started by circulating water at 90°F. The temperature of the circulating water was then raised at different rates -

1/

1, 2, 3, 4 and 5°F/hour - in order to give various rates of sweating. The sweating was stopped at the same cut out setting point in the strainings (108°F) in every test. At the slowest rate (1.4%/hour - Test No.20) the time for sweating was 21 hours, and 70.3% of single sweat wax of oil content 7.2% was obtained. At the fastest rate (4.9%/hour - Test No.24) the sweating time was only 6 hours, and the yield of single sweat wax was 70.4%. This wax contained 8.2% oil.

Temperature at start of sweating.

In the second series of tests the temperature to which the charge was cooled was varied, and also the temperature of the circulating water at start of sweating. The temperature was raised at the rate of 2°F/hour in each test. In Test No.25 the charge was cooled to 70°F. and circulating water pumped at 70°F. to start sweating. The time for sweating was 22½ hours and the single sweat wax amounted to 73.0% and contained 7.9% oil. Due to the low temperature at the start of the sweating 6.2% of residue oil was collected. The temperature to which the wax was cooled was gradually increased to 105°F. in Test No.28. In this test wax was cooled to 105°F. and sweating commenced by pumping water at 105°F. This procedure reduced the time for sweating to only 3¾ hours, and a yield of 59.9% single sweat wax was obtained. The single sweat wax had an oil content of 7.6%.

In/

In Test No.29 the charge was cooled to 95°F. and sweating started by pumping water at 95°F. The temperature of the circulating water was raised 3°F/hour till finish of sweating. The time of sweating under these conditions was 6½ hours, and a yield of single sweat wax of 68.4% having an oil content of 6.8% was realised.

Discussion of Results.

In order to decide which of the above conditions were the best the products percentage on charge per hour (cooling and sweating) was calculated for each test. In Tests 25 and 26 the residue oil fraction collected at start of sweating was considered as a product since this does not require to be recycled. The details of all tests including the products percentage on charge per hour are given in Table 15.

Test No.20 gave the lowest yield per hour, 2.73%, while Test No.28 gave the highest, 8.56%. In the latter test the charge was cooled to 105°F., the cooling time being 3¼ hours and sweating time only 3¾ hours. The test which gave the second highest yield per hour (6.51%) was Test No.29. The charge in this case was cooled to 95°F and sweating commenced by pumping water at the same temperature. The temperature was raised 3°F/hour. Under these conditions the cooling time was 4 hours and sweating 6½ hours. For operations on a large scale it was deemed that the conditions applied in this test would be preferable to/

to those of Test No.28. When cooling to 105°F. the yields for the individual runs were more erratic, and the rate of sweating at the start was uncertain, part of the charge in some of the tests being not sufficiently cooled.

In Table 15 are also given the results of sweating a similar charge in a Henderson House. For this plant cooling required 30 hours and sweating 28 hours. The yield of single sweat wax was 69%, the oil content being 6.4%. For the Henderson House the products percentage on charge per hour was only 1.2%. A comparison of the two plants when sweating hard scale is given in Table 14.

TABLE 14

		Experimental Sweater	Henderson House
Test No.		29	30
Charge:		Hard scale	Hard scale
Setting Point,	°F.	120	120
Oil content,	%	18.6	18.6
Times:			
Cooling,	hours,	4	30
Sweating,	"	6½	28
Total	"	10½	58
Yields:			
1st Strainings,	%	31.6	31.0
Single Sweat Wax,	%	68.4	69.0
Analyses of Products:			
1st Strainings.			
Setting Point,	°F.	101.0	98.0
Oil content,	%	47.1	51.2
Single Sweat Wax.			
Setting Point,	°F.	126.3	126.0
Oil content,	%	6.8	6.4

(b)/

TABLE 15

SWEATING HARD SCALE TO SINGLE SWEAT WAX IN EXPERIMENTAL SWEATER UNDER DIFFERENT CONDITIONS AND IN HENDERSON HOUSE UNDER NORMAL CONDITIONS.

	EXPERIMENTAL SWEATER										HENDERSON HOUSE
Test No:	20	21	22	23	24	25	26	27	28	29	30
Charge:	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale
Gallons,	300	305	300	296	294	290	286	303	280	284	5700
Temperature, °F.	141	148	140	143	145	145	153	151	146	144	150
Setting Point, °F.	120	120	120	120	120	120	120	120	120	120	120
Oil content, %	18.6	18.6	18.6	18.6	18.6	18.6	18.6	18.6	18.6	18.6	18.6
Times:											
Cooling, hours	4¾	4¾	4½	5	5	7½	7½	4	3¼	4	30
Sweating, "	21	11¾	9¼	7	6	22½	18¼	8¼	3¾	6½	28
Total "	25¾	16½	13¾	12	11	30	25¾	12¼	7	10½	58
Yields: % on charge,											
Residue Oil,	-	-	-	-	-	6.2	5.4	-	-	-	-
1st Strainings,	29.7	30.5	29.2	28.9	29.6	20.8	22.8	30.6	40.1	31.6	31.0
Single Sweat Wax,	70.3	69.5	70.8	71.1	70.4	73.0	71.8	69.4	59.9	68.4	69.0
Single Sweat Wax + Res.Oil % on charge per hour (cooling and sweating)	2.73	4.21	5.15	5.93	6.40	2.64	3.00	5.67	8.56	6.51	1.2
Conditions:											
Temp.to which charge cooled, °F.	85	85	85	85	85	70	75	95	105	95	-
Temp.of water at start of sweating, °F.	90	90	90	90	90	70	80	95	105	95	-
Temp.of house at start of sweating, °F.	-	-	-	-	-	-	-	-	-	-	88
Max.temp.of water during sweating, °F.	111	113.5	117	118	120	115	116.5	111.5	112.5	114	-
Max.temp.of house during sweating, °F.	-	-	-	-	-	-	-	-	-	-	119
Rate of increase in temp. °F/hour,	1	2	3	4	5	2	2	2	2	3	2 to max.
Sett.Pt.at cut out, °F.	108	108	108	108	108	108	108	108	108	108	108
Rate of Sweating: % per hour on charge,	1.4	2.6	3.2	4.1	4.9	1.2	1.5	3.7	10.7	4.9	1.1
Analyses of Products:											
1st Strainings.											
Setting Point, °F.	99	99	101	100	101	101	99	100	106	101	98
Oil content, %	52.3	50.1	51.1	56.9	54.3	50.1	50.9	49.4	41.8	47.1	51.2
Single Sweat Wax.											
Setting Point, °F.	126.5	126.5	126.4	125.8	126.0	126.3	126.6	126.6	127.4	126.3	126.0
Oil content, %	7.2	6.6	8.1	7.6	8.2	7.9	7.4	7.3	7.6	6.8	6.4

(b) Sweating Single Sweat Wax to Wax of Oil content 0.5%

Single sweat wax prepared at the Henderson Houses from hard scale was sweated to give wax having an oil content of about 0.5% in the experimental sweater, to find the best conditions of operation. The same single sweat wax was sweated in the Henderson House under normal conditions, and the optimum conditions for the tube sweater having been found, a comparison between the two sweaters was made.

The single sweat wax used had a setting point of 126.4°F. and oil content 5.0%.

In the Henderson House (Test No.42) the rate of sweating was 0.8% per hour on charge, and the yield of wax was 49% on charge. The wax had a setting point of 131.4°F. and oil content 0.5%.

The experimental sweater was operated to begin with at a rate of sweating similar to the Henderson House, and the conditions of sweating then varied to give increased rates. In all, eleven different sets of conditions were investigated.

Rate of raising temperature of circulating Water.

In the first tests (Nos.31-34) the charge was cooled to 105°F. and sweating started by pumping water at a temperature of 110°F. The water temperature was raised to a maximum of 127°F. at different rates. By raising the temperature/

temperature ½°F/hour to the maximum, the time for sweating was 68 hours, by raising at 1°F/hour the time was 34½ hours, at 2°F/hour the time was 27¼ hours, and at 3°F/hour the time was reduced to 22¼ hours. The yield of wax at the slowest rate was 47.6% , and at the fastest rate 47.4%. In the other two tests the yields were 52.5% and 49.7%. The waxes ranged in setting point from 131.1°F. to 131.6°F. and oil content from 0.3% to 0.5%.

Maximum temperature of circulating water.

The effect of heating the circulating water to a higher maximum temperature was investigated (Tests 35 and 36). The temperature was raised 3°F/hour to a maximum of 129°F. and to 131°F., all other conditions being the same as before. Increasing the temperature of the water to 129°F. gave a sweating time of 22¼ hours, while a maximum of 131°F. gave a time of 17¼ hours. The yield of wax in Test No.35 was 38.5% and in Test No.36 33.3%.

Temperature of circulating water at start of sweating.

Since it appeared that raising the water temperature above 127°F. considerably reduced the yield of wax, it was decided to fix on 127°F. as a maximum, and the starting temperature was then raised from 110°F. to 120°F. (Test No.37). Under these conditions the sweating time was 18 hours, and wax 45.9% on charge, with a setting point of 131°F. and oil content 0.3% was obtained.

Temperature to which charge cooled.

In test No.38 the sweating time was reduced to 16 hours by raising the temperature to which the wax was cooled from 105°F. to 110°F. The wax - 45% on charge - had a setting point of 130.7°F. and oil content 0.5%.

The temperature to which the wax was cooled was further increased to 115°F. with the result that the sweating time was reduced to 14 hours. The yield of wax in this case was 47.3% on charge, the oil content being 0.3%

By raising the temperature to which the wax was cooled to 120°F. (Test No.40), the time necessary for sweating was reduced to 12 hours, but the yield of wax of the desired quality was reduced to 41.1%.

As a matter of interest a last test was made in which the charge was cooled to 123°F. and sweating commenced with water at 123°F. In this test (No.41) the sweating time was only 10 hours, but there was a further drop in yield. The wax amounted to 32.8% on charge, with a setting point of 131.2°F. and oil content 0.3%.

Discussion of Results.

The details of these tests are given in Table 17, and by studying this it will be seen how important it is to have accurate control of the temperature conditions. For example, by raising the maximum temperature from 127°F. to 129°F. the wax yield fell from 47% to 38%, and by raising the temperature to 131°F. the yield was only 33%./

33%. By cooling to 115°F. instead of 105°F. and by starting to sweat with water at 120°F. instead of 110°F. the time for cooling and sweating was reduced from 25¼ hours to 16½ hours without any decrease in yield of product.

Two tests (Nos.36 and 39) having the same average rate of sweating gave very different yields. In the former test the charge was cooled to 105°F., sweating started with water at 110°F. and the maximum temperature 131°F., whereas in Test No.39 the charge was cooled to 115°F., water at start of sweating being 120°F. and the maximum temperature 127°F. In Run 36 the yield of wax was only 33% whereas inRun 39 the wax amounted to 47%, and therefore sweating should be slow at the finish and fast at the beginning. This point is demonstrated in Fig.7, which shows the rates of sweating under different conditions during these tests.

In tests 34, 35 and 36 the charge was cooled to 105°F. and from Fig.7 it will be seen that the sweating rate at the beginning was low and increased to a maximum after about 7 hours. In Test 39 the temperature to which the charge was cooled was 115°F. and in this case the rate of sweating was a maximum at the start then gradually decreased. The rate of sweating at the start was 15% on charge per hour. In Tests 40 and 41 the charge was cooled to 120°F. and 123°F. respectively, and again/

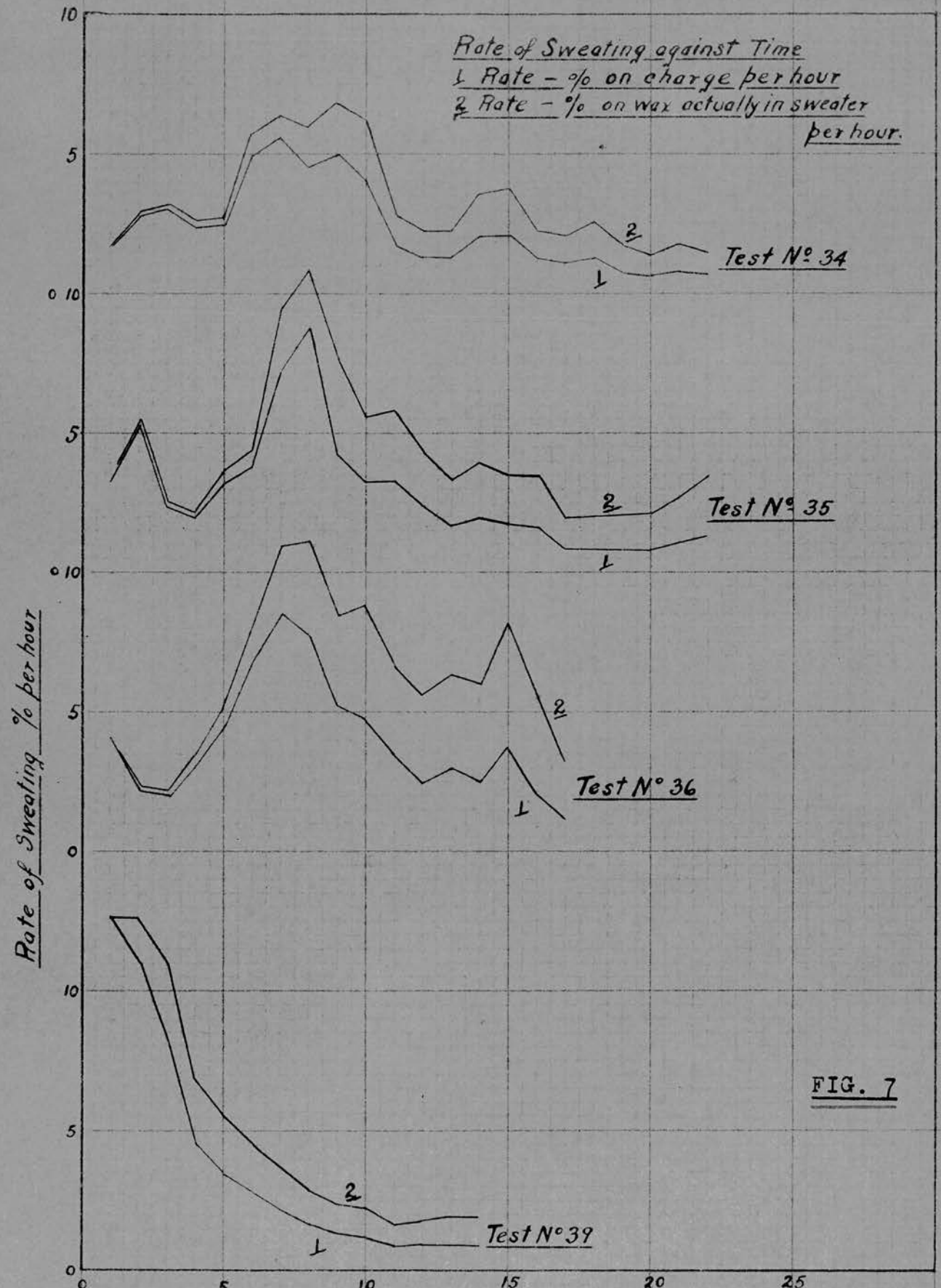

Rate of Sweating against Time
1 Rate – % on charge per hour
2 Rate – % on wax actually in sweater per hour.
Test No 34
Test No 35
Test N° 36
Test N° 39
Rate of Sweating % per hour
Time in hours
0
5
10
15
20
25
10
5
0

FIG. 7

again the maximum rate was at the start of the operation (see Fig.8) but in this case the sweating was very fast, being 31% in Test No.40 and 50% in No.41. It will be remembered that in both of these tests there was a definite fall in yield, and this must have been due to the charge being insufficiently cooled. The tests (Nos.34, 39, 40 and 41) in which the maximum temperature was 127°F. showed low rates of sweating towards the finish of the period, the average over the last 4 hours being 0.8% on charge per hour. With a maximum temperature during sweating of 129°F. the rate for the last four hours was 1.1% per hour, the corresponding figure for a maximum temperature of 131°F. being 2.2% per hour.

Even the graph showing rate of sweating percentage per hour on charge against time does not show the true rate of sweating, because the quantity of wax in the sweater decreases during the operation and the true measure of rate of sweating should be percentage per hour. on wax actually in the sweater. This is important since the rate at the finish should be slow. If the yield of wax is say 50% on charge, then by expressing the rate as percentage per hour on charge the figure at the finish will be only half the true rate of sweating. In Figs.7 and 8 the true rates of sweating, that is rate percentage per hour on wax actually in sweater, are also given. With a maximum temperature of 127°F. the average true rate of sweating/

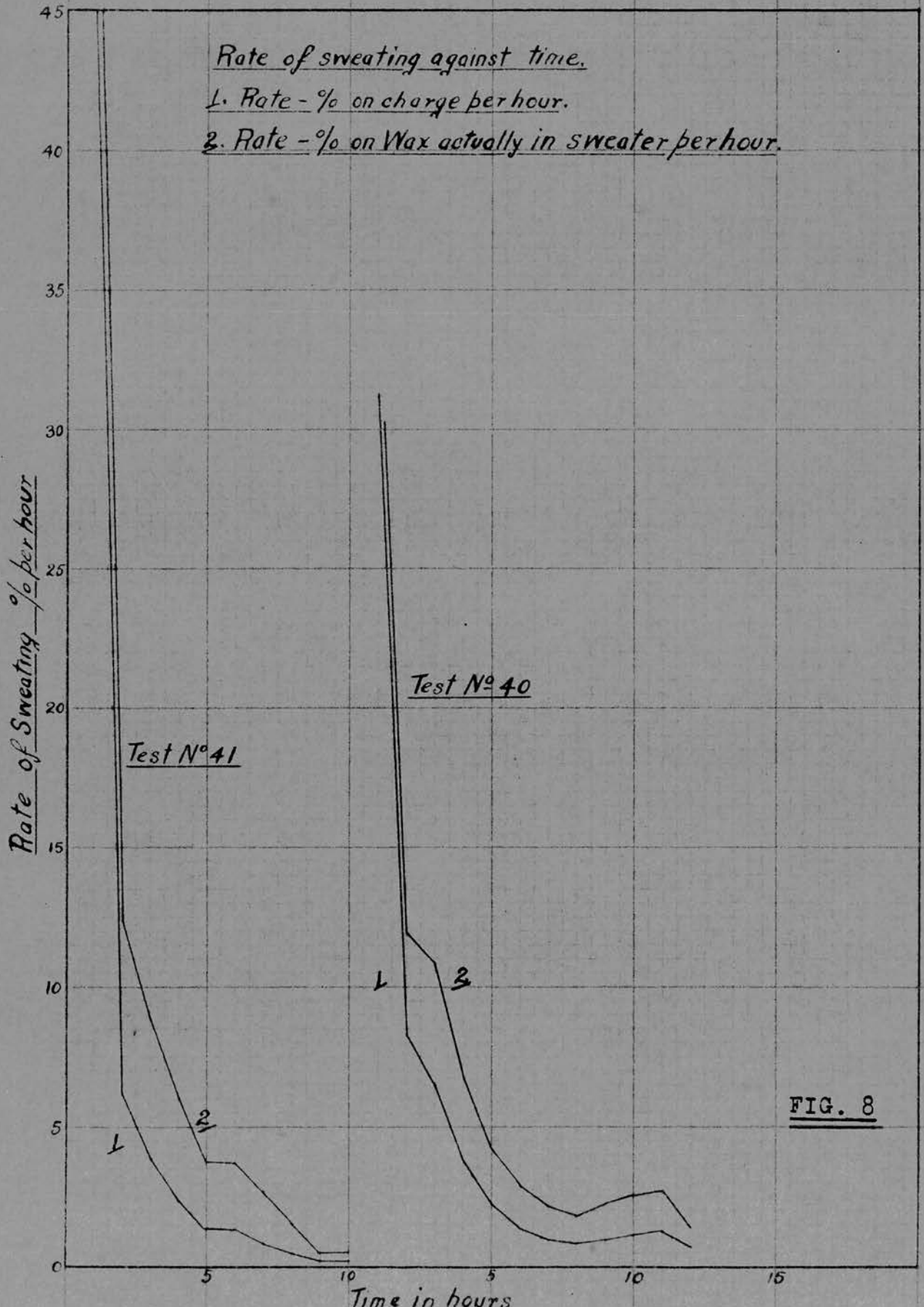

Rate of sweating against time.
1. Rate - % on charge per hour.
2. Rate - % on Wax actually in sweater per hour.
Rate of Sweating - % per hour
Time in hours
Test N° 41
Test N° 40
1
2
1
2
45
40
35
30
25
20
15
10
5
0
5
10
5
10
15

FIG. 8

sweating during the last four hours was 1.7% per hour, while with a maximum temperature of 129°F. the true rate was 2.6% per hour. When the maximum temperature was raised to 131°F. the corresponding rate of sweating was 6.0% per hour. In the tests in which the maximum temperatures were 129°F. and 131°F. the yield of wax was considerably lower than with a maximum temperature of 127°F.

As in the case of the hard scale experiments, in order to decide the optimum conditions for sweating, the percentage products/hour (cooling and sweating) has been calculated. This is given in Table 17 together with details of all tests. The yield, percentage per hour, varied from 0.67 to 2.86. The highest rate (2.86%) was obtained in Run 39, which gave an average rate of sweating of 3.8%. The Henderson House results showed 0.53% product/hour, and a rate of sweating of 0.8% per hour.

The best conditions for the experimental sweater were therefore to cool to 115°F., start sweating with water at 120°F. and raise at the rate of 3°F/hour to a maximum of 127°F.

A comparison of the experimental sweater under these conditions and the Henderson House is given in Table 16.

TABLE 16/

TABLE 16

		Experimental Sweater	Henderson House
Test No.,		39	42
Charge:		Single Sweat Wax	Single Sweat Wax.
Setting Point,	°F.	126.4	126.4
Oil content,	%	5.0	5.0
Times:			
Cooling,	hours,	2½	25
Sweating,	"	14	67
Total	"	16½	92
Yields:	% on charge,		
3rd Strainings,		52.7	51.0
Wax,		47.3	49.0
Analyses of Products:			
3rd Strainings.			
Setting Point,	°F.	123.0	121.0
Oil content,	%	5.6	7.6
Wax.			
Setting Point,	°F.	131.1	131.4
Colour (6" cell)		6.5Y+1.4R	5.0Y+1.2R
Oil content,	%	0.3	0.5
Hardness No. at 90°F.		8.2	8.0

(c) Comparison of Double Sweating and Single Sweating of Hard Scale

In the tests already described hard scale was sweated to single sweat wax, and single sweat wax was resweated to wax of setting point about 131°F. containing approximately 0.5% of oil. This process, termed double sweating, is generally applied to fractions which would give a low yield of finished products if sweating was done in one stage. It is generally considered that the time lost in melting out, recharging and cooling is compensated for by the fact that on recharging the sweater is filled to capacity/

TABLE 17

SWEATING SINGLE SWEAT WAX IN THE EXPERIMENTAL SWEATER UNDER DIFFERENT CONDITIONS AND IN THE HENDERSON HOUSE UNDER NORMAL CONDITIONS.

	EXPERIMENTAL SWEATER											HENDERSON HOUSE
Test No:	31	32	33	34	35	36	37	38	39	40	41	42
Charge:	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax	Single Sweat Wax
Gallons,	298	280	278	271	315	280	280	287	287	280	305	4025
Temperature, °F.	153	170	161	155	155	156	145	153	154	151	154	146
Setting Point, °F.	126.4	126.4	126.4	126.4	126.4	126.4	126.4	126.4	126.4	126.4	126.4	126.4
Oil content, %	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Times:												
Cooling, hours,	3	3	3	3	3	3	3	3	2½	2½	2	25
Sweating, "	68	34½	27¼	22¼	22¼	17¼	18	16	14	12	10	67
Total "	71	37½	30¼	25¼	25¼	20¼	21	19	16½	14½	12	92
Yields of Products:												
% on charge,												
3rd Strainings,	52.4	47.5	50.3	52.6	61.5	66.7	54.1	55.0	52.7	58.9	67.2	51.0
Wax,	47.6	52.5	49.7	47.4	38.5	33.3	45.9	45.0	47.3	41.1	32.8	49.0
Wax, % on chg./hour (cooling and sweating)	0.67	1.40	1.64	1.88	1.52	1.64	2.19	2.37	2.86	2.83	2.73	0.53
Conditions:												
Temp.to which charge cooled, °F.	105	105	105	105	105	105	105	110	115	120	123	-
Temp.of water at start of sweating, °F.	110	110	110	110	110	110	120	120	120	120	123	-
Temp.of house at start of sweating, °F.	-	-	-	-	-	-	-	-	-	-	-	90
Max.temp.of water during sweating, °F.	127	127	127	127	129	131	127	127	127	127	127	-
Max.temp.of house during sweating, °F.	-	-	-	-	-	-	-	-	-	-	-	130
Rate of increase in temp. °F/hour,	½ to max,	1 to max,	2 to max,	3 to max,	3 to max,	3 to max.	3 to max,	3 to max,	3 to max.	3 to max,	3 to max,	3 to max.
Setting Pt.at cut out, °F.	125½	125½	125½	125½	125½	127.3	125½	125½	125½	125½	125½	125
Rate of Sweating.												
% per hour on charge,	0.8	1.4	1.8	2.3	2.8	3.8	3.0	3.4	3.8	4.9	6.7	0.8
Analyses of Products:												
3rd Strainings.												
Setting Point, °F.	122	122	122	122	124	125	123	123	123	124	125	121
Oil content, %	7.0	8.0	6.8	6.4	4.8	6.0	6.8	6.4	5.6	6.0	5.6	7.6
Wax.												
Setting Point, °F.	131.6	131.1	131.1	131.1	131.5	131.3	131.0	130.7	131.1	131.5	131.2	131.4
Colour (6" cell),	7.0Y+ 1.6R	8.5Y+ 1.8R	7.5Y+ 1.5R	7.0Y+ 1.6R	7.5Y+ 1.7R	8.5Y+ 1.9R	8.5Y+ 1.8R	8.5Y+ 1.8R	6.5Y+ 1.4R	7.0Y+ 1.6R	7.0Y+ 1.6R	5.0Y+ 1.2R
Oil content, %	0.5	0.5	0.5	0.3	0.5	0.5	0.3	0.5	0.3	0.2	0.3	0.5
Hardness No. at 90°F.	9.8	7.2	8.4	9.1	8.4	8.2	7.4	6.5	8.2	9.8	8.8	8.0

capacity, and so long times with small quantities of wax in the sweater are avoided.

During discussions on the merits of sweaters, a question which is nearly always asked is "can the sweater give a finished wax in one sweat?". It was considered therefore that an investigation should be made to find if double sweating has advantages over sweating direct to finished wax in one step, and if so, when it should be applied.

Tests were made both in the experimental sweater and in the Henderson House in which wax of setting point about 131°F. was prepared from hard scale by double sweating and single sweating, that is, sweating direct to desired wax without melting out and recharging. In each comparison the conditions aimed at were such that the only change was the double or single sweating.

Tests on Experimental Sweater.

In Test No.43 the charge (hard scale) had a setting point of 118°F. and oil content 20.9%. This was sweated to single sweat wax of setting point 125.3°F. and oil content 4.3%. The conditions for this sweating were to cool to 95°F., start sweating with water at 95°F. and raise the temperature of the water 3°F/hour. The sweating was stopped when the strainings leaving the sweater were of setting point 108°F. This gave a yield of single/

single sweat wax of 66.8%. The time for cooling was 5¾ hours and for sweating 8 hours.

When resweating, the single sweat wax was cooled to 115°F. and sweating commenced with water at 120°F., the temperature then being raised at the rate of 3°F/hour to a maximum of 127°F. The sweating was stopped when the strainings leaving the plant had a setting point of 125.5°F. The cooling took 2 hours and sweating 14 hours. The yield of double sweat wax was 39% on charge, which is 26.0% on hard scale. The wax had a setting point of 131.7°F. and oil content 0.3%.

The same hard scale was then sweated (Test No.44) to give wax of approximately the same quality as the double sweat wax. The charge was cooled to 95°F. sweating started with water at 95°F., the temperature being raised 3°F/hour to a maximum of 127°F. The strainings up to a setting point of 108°F., correspond with first strainings, and subsequent strainings were collected separately to compare with third strainings from single sweat wax. The sweating was continued for 22 hours, which was the total actual sweating time applied to double sweat wax. The strainings setting point at the finish of the sweating was 125.5°F., so that the wax in the sweater had exactly the same treatment as the double sweat wax except that it had/

had not been melted out and recharged.

The sweated wax had a setting point of 130.1°F. and oil content 1.0%, and the yield was 29.6% on hard scale. This is a higher yield, but a poorer quality than under the double sweating conditions.

Test No.44 was then repeated under exactly the same conditions except that the circulating water maximum temperature was raised from 127°F. to 128°F. and sweating was continued for three hours longer. The product in Test No.45 had a setting point of 130.4°F. and oil content 0.7%, and the yield was 24.6% on hard scale. This yield is lower than that of test No.43 (double sweating) and the quality is still poorer. The fact that the product had a lower setting point and higher oil content than the double sweat wax in spite of the longer sweat and at a higher temperature, shows that melting and recharging actually make sweating easier and have resulted in a slightly greater yield and a better product.

The tests are summarised in Table 18.

The times shown for sweating under the different conditions are not exactly comparable. The time for sweating single sweat wax should be reduced since the quantity of single sweat wax from a charge of hard scale would not completely fill the sweater. Also, in double sweating, the stove has to be filled twice, and melting out takes place twice, and in the calculations given/

given in Table 19, to give comparable times, the times for charging and melting out are those for a 20 ton sweater, so that the conclusions would apply to a plant of that size.

TABLE 18

	Double Sweating	Single Sweating	
Test No.	43	44	45
Charge:	Hard Scale	Hard Scale	Hard Scale
Setting Point, °F.	118	118	118
Oil content, %	20.9	20.9	20.9
Times:			
Cooling hard scale, hrs.	5¾	5¾	5¾
" Single Sw.Wax,"	2	-	-
Total cooling, "	7¾	5¾	5¾
Sweating hard scale, "	8	22	25¼
" Single Sw.Wax, "	14	-	-
Total sweating, "	22	22	25¼
Total time, "	29¾	27¾	31
Yields: % on hard scale.			
1st Strainings,	33.2	32.8	32.7
3rd Strainings,	40.8	37.6	42.7
Wax,	26.0	29.6	24.6
Analyses of Sweated Wax.			
Setting Point, °F.	131.7	130.1	130.4
Oil content, %	0.3	1.0	0.7
Colour, (6" cell)	6.0Y+ 1.2R	8.7Y+ 1.9R	8.5Y+ 1.8R

TABLE 19

	Double Sweating.	Single Sweating.	
Test No:	43	44	45
Sweating Hard Scale.			
Charging, hours,	1	1	1
Cooling, "	5¾	5¾	5¾
Sweating, "	8	22	25¼
Running out, "	4	3	3
	18¾	31¾	35
Sweating Single Sweat Wax.			
Charging, hours,	1		
Cooling, "	2		
Sweating, "	14		
Running out, "	3		
	20		
Total cooling, hours,	7¾	5¾	5¾
" sweating, "	22	22	25¼
" time, "	38¾	31¾	35

The time to deal with one ton of hard scale

in/

in a one ton sweater under double sweating conditions would be:-

Sweating 1 ton hard scale - 1 x 18.75	= 18.75 hrs.
Sweating .668 ton (yield of S.S.Wax x 20 on hard scale)	= 13.4 "
	32.15 "

When dealing with one ton of hard scale by single sweating in Test No.44 the time was 31¾ hours, and 35 hours in Test No.45. This means that in Test No.45 the time by single sweating was greater than by double sweating, and the yield was poorer by single sweating - 24.6% on hard scale as against 26.0%. At the same time the oil in the wax by single sweating was 0.7% against 0.3% by double sweating.

The above experiments make it clear that double sweating is a definite advantage over single sweating, and it should be possible to calculate the yield to which the first sweating should be taken in order to give the minimum time for the complete operation. This has been arrived at by calculating the time necessary for the first and second sweatings for different yields of single sweat wax using the test times as a basis. An example is given below:-

Assume yield of single sweat wax = 60% on hard scale.
i.e. 6.8% less than in test. Av.rate of sweating = 4.3%/hr.
∴ 1½ hours longer sweating for hard scale and 1½ hours less for second sweating.

Sweating hard scale	= 20¼ hours
Sweating Single Sw.Wax	= 18½ "

Corrected/

Corrected time for complete operations would be:-

1 x 20.25	=	20.25 hours
.6 x 18.5	=	11.1 "
		31.35 "

Times to completely sweat hard scale calculated for different yields of single sweat wax are as under:-

TABLE 20

Yield of Single Sweat Wax. % on hard scale.	Total time to sweat. hours.
70	32.5
66.8	32.1
60	31.3
55	31.0
50	30.7
45	30.3
40	30.5

These results show that for the experimental sweater the time for sweating is a minimum when the first charge is sweated to about a 45% yield before running out and recharging. There is very little difference in total time, however, over a wide range of yields.

Details of the tests are given in Table 23.

Tests on Henderson House.

Tests similar to those made on the tube sweater have been carried out on the Henderson House. As in the case of the previous tests care was taken that conditions were exactly the same except melting out and recharging. Table 21 summarises the results.

TABLE 21/

TABLE 21

	Double Sweating.	Single Sweating.
Test No.	46	47
Charge:	Hard scale	Hard scale
Setting Point, °F.	117.9	117.9
Oil content, %	14.9	14.9
Times:		
Sweating Hard Scale.		
Charging, hours,	1	1
Cooling, "	25	25
Sweating, "	28½	71
Running out Single Sw.Wax,	4	4
	58½	101
Sweating Single Sweat Wax.		
Charging, hours,	1	
Cooling, "	20	
Sweating, "	30½	
Running out sweated wax,	3	
	54½	
Total cooling, hours,	45	25
" sweating, "	59	71
" time "	113	101
Yields: % on hard scale.		
1st Strainings,	26.8	31.7
3rd "	40.3	37.2
Wax,	32.9	31.1
Analyses of Sweated Wax.		
Setting Point, °F.	131.0	131.1
Oil content, %	0.8	0.8
Colour (6" cell)	9.0Y+1.6R	10.2Y+2.3R

The total time to process one charge of hard scale by double sweating would be:-

Sweating hard scale,	58.5 hours
Sweating .732 ton Single Sweat wax for 54.5 hrs.	39.8 "
Total,	98.3 "

The time by double sweating for the complete operation was therefore 98.3 hours against 101 hours for single sweating. The yield of wax by double sweating was higher/

higher than by single sweating - 32.9% compared with 31.1%, while the waxes had very similar setting points and oil contents. The colour of the double sweated product was slightly lower than that of the wax made by single sweating.

The times for double sweating at different yields of single sweat wax were calculated as in the case of the tube sweater, and the results are given in Table 22.

TABLE 22

Yield of Single Sweat Wax. % on hard scale.	Total time for sweating. hours.
80	101.1
73.2	98.1
70	97.4
60	95.1
55	94.5
50	94.3
45	94.6
40	95.0

The time for double sweating is therefore a minimum when the yield of single sweat wax is 50% on charge, but as in the case of previous tests the yield of single sweat wax over a wide range has very little effect on the total time.

Particulars of Tests 46 and 47 are given in Table 23.

Conclusions.

The conclusions for the comparative tests made in both sweaters are that double sweating resulted in a slightly higher yield and a slightly better wax than sweating direct to finished wax without melting out and recharging/

TABLE 23

COMPARISON OF DOUBLE SWEATING AND SINGLE SWEATING OF HARD SCALE IN EXPERIMENTAL SWEATER AND IN HENDERSON HOUSE.

	EXPERIMENTAL SWEATER			HENDERSON HOUSE		
Test No:	43 Double Sweating		44 Single Sweating	45 Single Sweating	46 Double Sweating		47 Single Sweating
Charge:	Hard Scale	Single Sweat Wax	Hard Scale	Hard Scale	Hard Scale	Single Sweat Wax	Hard Scale
Gallons,	294	281	290	289	5774	4140	4287
Temperature, °F.	147	148	150	153	130	150	140
Setting Point, °F.	118	125.3	118	118	117.9	123.4	117.9
Oil content, %	20.9	4.3	20.9	20.9	14.9	5.3	14.9
Times:							
Cooling, hours	5¾	2	5¾	5¾	25	20	25
Sweating, "	8	14	22	25¼	28½	30½	71
Total "	13¾	16	27¾	31	53½	50½	96
Yields: % on hard scale.							
1st Strainings,	33.2	-	32.8	32.7	26.8	-	31.7
3rd Strainings,	-	40.8	37.6	42.7	-	40.3	37.2
Wax,	66.8	26.0	29.6	24.6	73.2	32.9	31.1
Conditions:							
Temp.to which chg.cooled, °F.	95	115	95	95	-	-	-
Temp.of water at start of sweating, °F.	95	120	95	95	-	-	-
Temp.of house at start of sweating, °F.	-	-	-	-	90	103	96
Max.temp.of water during sweating, °F.	115	127	127	128	-	-	-
Max.temp.of house during sweating, °F.	-	-	-	-	120	131	131
Rate of increase in temp., °F/hour,	3 to max.	3 to max.	3 to max.	3 to max.	1.4 to max.	1.5 to max.	1.8 to max
Sett.Pt.at cut out,							
1st Strainings, °F.	108	-	108	108	106	-	106
3rd Strainings, °F.	-	125.5	125.5	125.5	-	125	125
Rate of Sweating.							
% per hr.on charge,	2.6	4.4	2.6	2.5	0.9	1.8	1.0
Analyses of Products.							
1st Strainings. Sett.Pt.°F.	101	-	98	103	97.7	-	96.7
Oil content %	47	-	52.7	42.8	44.7	-	45.8
3rd Strainings Sett.Pt.°F.	-	122	121.7	122.5	-	117	119
Oil content %	-	7.2	9.0	8.2	-	11.3	9.2
Single Sw.Wax. Sett.Pt.°F.	125.3	-	-	-	123.4	-	-
Colour(2"cell)	20Y+3.4R	-	-	-	17.5Y+4.7R	-	-
Oil content %	4.3	-	-	-	5.3	-	-
Wax. Sett.Pt.°F.	-	131.7	130.1	130.4	-	131.0	131.1
Colour (6"cell)	-	6.0Y+1.2R	8.7Y+1.9R	8.5Y+1.8R	-	9.0Y+1.6R	10.2Y+2.3R
Oil content %	-	0.3	1.0	0.7	-	0.8	0.8
Hardness No. at 90°F.	-	9.0	5.8	6.8	-	-	-

recharging. The times for the complete operation were very similar by the different methods.

It has been calculated that to give the minimum total time the first sweating should be continued until the yield of single sweat wax amounts to 45/50% on charge. At this stage the wax should be melted out and recharged. There is very little difference in total time, however, over a wide range of yields of single sweat wax.

(d) Sweating Hard Scales of Various Oil Contents.

In order to determine the effect of oil in scale on the yield of wax and time necessary for sweating, hard scales of different oil contents have been sweated to give waxes of oil contents about 0.5%. The same hard scale was used throughout the tests, the oil content being raised as required by the addition of wax-free oil.

The original hard scale had a setting point of 119°F. and contained 13.8% oil. Wax-free oil was added to this scale to give mixtures containing 20.9%, 26.1%, 33.7% and 56.7% oil. The original scale and each of the mixtures were then sweated to wax containing approximately 0.5% oil, the conditions in each case being such that the fall in yield of product was due entirely to the increase in oil in the charge. The results are summarised in Table 24.

TABLE 24/

TABLE 24

Test No.	48	49	50	51	52
Charge:	Hard Scale	Hard Scale	Hard Scale	Hard Scale	Hard Scale
Setting Point, °F.	119	118	116.5	113.5	107
Oil content, %	13.8	20.9	26.1	33.7	56.7
Sweating Time hours	25½	27	28½	30½	51
Yields: % on hard scale					
Strainings,	71.8	74.0	75.2	80.7	92.6
Wax,	28.2	26.0	24.8	19.3	7.4
Analyses of Wax.					
Setting Point, °F.	131.3	131.7	131.4	132.9	139.4
Oil content, %	0.6	0.3	0.5	0.8	0.5

Discussion of Results.

The yield of wax decreased from 28.2% to 7.4% as the oil in the charge was increased from 13.8% to 56.7%. As well as decreasing the yield, increasing the oil in charge necessitated longer sweating periods. The time increased from 25½ hours to 51 hours. In Test No.48, 25½ hours sweating gave 28.2% wax, while in Test No.52 51 hours sweating only resulted in 7.4% wax.

An interesting point observed was the increase in setting point of the wax as the oil in the charge increased. The wax from the lowest oil content charge had a setting point of 131.3°F. while the highest oil content charge gave a wax of setting point 139.4°F. Since the addition of oil to the charge lowered its setting point, this difference between setting point of charge and setting point of wax became very large as the oil increased.

TABLE 25/

TABLE 25

Oil in charge, %	13.8	20.9	26.1	33.7	56.7
Setting Pt.of charge, °F.	119	118	116.5	113.5	107
Setting Pt.of wax, °F.	131.3	131.7	131.4	132.9	139.4
Difference, °F.	12.3	13.7	14.9	19.4	32.4

The yield of wax which can be realised from scales of different oil content is shown graphically in Fig.9. The graph is a straight line and an important point demonstrated is that at 70% oil in charge no wax can be obtained by sweating. When sweating any fraction to give residue oil, the cut out from residue oil should therefore be the point at which the sweatings contain 70% oil.

All oil charged in the scale to a sweating plant must eventually go into residue oil, and since the oil content of a very large number of residue oil samples was found to be 71.6%, it is possible to calculate the total yield of wax which could be expected from scale of any oil content. By total yield of was is meant the wax made by direct sweating and by sweating strainings completely to residue oil and wax. A curve showing the effect of oil on totalyield of wax is also given in Fig.9. A few points from the curve are given in Table 26.

A study of the two graphs shows that the total quantity of wax which can be prepared from any charge is about 2.8 times the quantity which can be made from the same charge by direct sweating, i.e. without resweating/

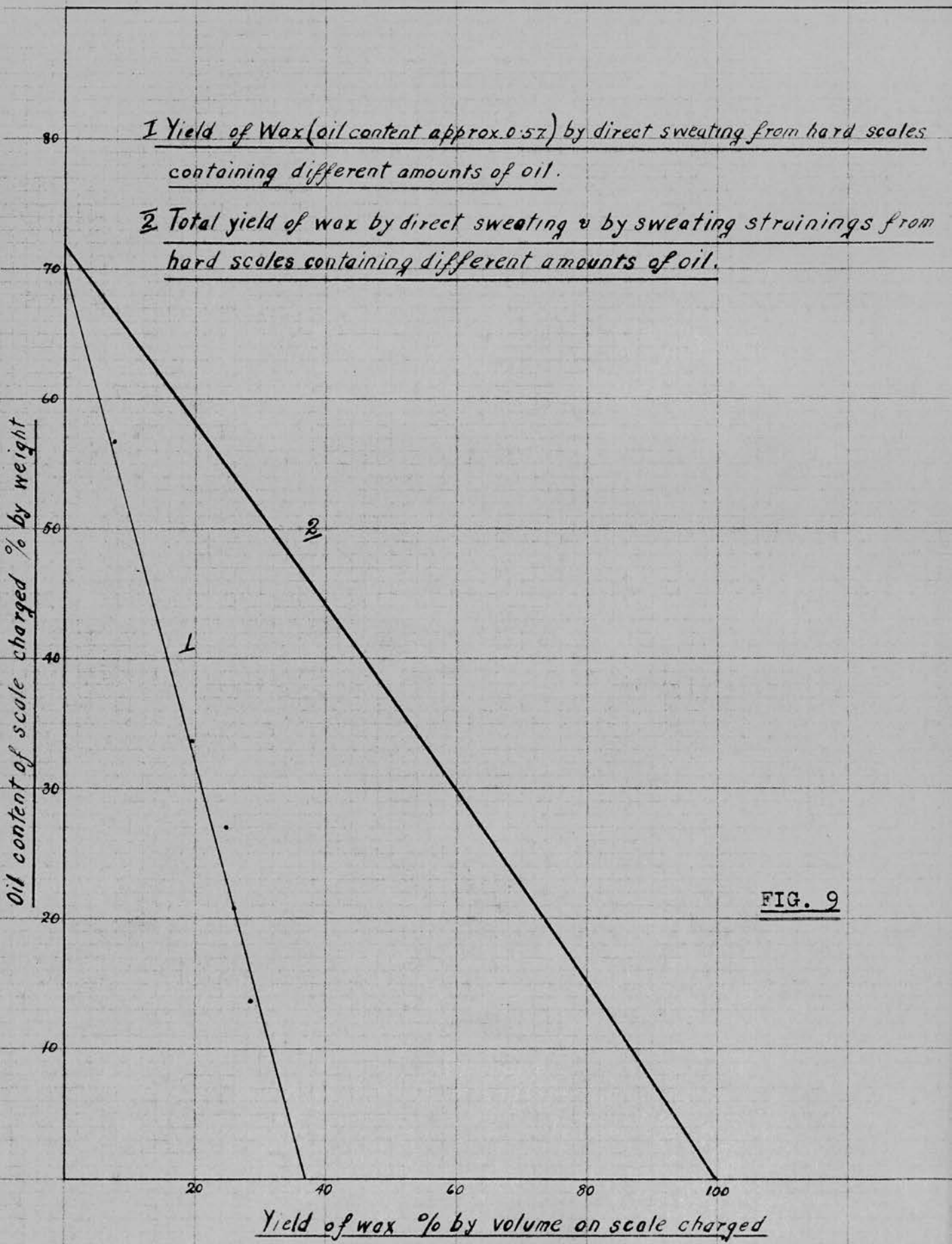

1 Yield of Wax (oil content approx. 0·5%) by direct sweating from hard scales containing different amounts of oil.
2 Total yield of wax by direct sweating & by sweating strainings from hard scales containing different amounts of oil.
Oil content of scale charged % by weight
80
70
60
50
40
30
20
10
1
2
20
40
60
80
100
Yield of wax % by volume on scale charged

FIG. 9

resweating any of the strainings.

TABLE 26

Oil in Scale % by wt.	Total Yield of Wax % by volume
10	86
20	73.5
30	60
40	46
50	32
60	17.5

The results of the tests demonstrate the importance of keeping the oil in the scales charged to the sweating plant at a minimum because of the decrease in yield of wax and the increased sweating to be done. At the same time the oil content of the charge must be known in order that the necessary difference in setting point between charge and wax is obtained, and suitable sweating conditions are applied.

Details of the conditions during the various tests are given in Table 27.

(e) Sweating First Strainings.

First strainings from hard scale is normally one of the oiliest fractions dealt with, and the bulk of the oil in hard scale is eliminated from the sweating plant when this fraction is sweated. Tests were therefore made to find how the tube sweater would deal with such an oily charge.

First strainings, setting point 103°F. and oil content 37.3%, were sweated at various rates to give residue oil, second strainings and a single sweat

wax/

TABLE 27

SWEATING HARD SCALES OF VARIOUS OIL CONTENTS IN EXPERIMENTAL SWEATER

Test No:	48		49		50		51		52		
	Hard Scale oil content 13.8%		Hard Scale oil content 20.9%		Hard Scale oil content 26.1%		Hard Scale oil content 33.7%		Hard Scale oil content 56.7%		
Charge:	Hard Scale	Single Sweat Wax	Hard Scale	Single Sweat Wax	Hard Scale	Single Sweat Wax	Hard Scale	Single Sweat Wax	Hard Scale	Single Sweat Wax	Double Sweat Wax
Gallons,	275	281	294	281	292	270	291	271	295	253	276
Temperature, °F.	150	151	147	148	149	151	146	151	149	149	161
Setting Point, °F.	119	125	118	125.3	116.5	126.2	113.5	127.4	107	120.8	134.5
Oil content, %	13.8	5.1	20.9	4.3	26.1	4.6	33.7	6.6	56.7	16.8	3.2
Times:											
Cooling, hours,	2½	2¼	5¾	2	5½	2	4¾	1½	3½	2½	1½
Sweating, "	11½	14	13	14	12½	16	12½	18	16	18	17
Total "	14	16¼	18¾	16	18	18	17¼	19½	19½	20½	18½
Yields: % on hard scale											
Strainings,	33.9	37.9	33.2	40.8	39.7	35.5	50.0	30.7	67.7	18.6	6.3
Wax,	66.1	28.2	66.8	26.0	60.3	24.8	50.0	19.3	32.3	13.7	7.4
Conditions:											
Temp.to which charge cooled, °F.	85	115	85	115	85	115	85	115	85	102	120
Temp.of water at start of sweating, °F.	90	120	90	120	90	120	90	120	90	105	125
Max.temp.of water during sweating, °F.	112	127	115	127	115	127	115	127	118	124	136
Rate of increase in gemp., °F/hour,	2	3 to max.	2	3 to max.	2	3 to max.	2	3 to max.	2	3 to max.	3 to max.
Sett.Pt.at cut out, °F.	108	125.5	108	125.5	108	125.5	108	125.5	110	121	133.5
Rate of Sweating. % per hr. on charge,	3.0	3.6	2.6	4.4	3.2	3.7	4.3	3.5	4.2	3.5	2.7
Analyses of Products:											
Strainings.											
Setting Point, °F.	102.1	120.0	101	122	98	121.5	94.5	122.5	99	110	128.3
Oil content, %	36.4	8.0	47.0	7.2	59.9	8.8	63.5	12.0	65.5	48.8	5.0
Wax.											
Setting Point, °F.	125.0	131.3	125.3	131.7	126.2	131.4	127.4	132.9	120.8	134.5	139.4
Oil content, %	5.1	0.6	4.3	0.3	4.6	0.5	6.6	0.8	16.8	3.2	0.5

wax containing about 5% oil. The same charge was processed in a Henderson House so that a comparison between the experimental sweater and the Henderson House could be made.

In Tests 53-58 the charge was cooled to 70°F. (3-3½ hours) and sweating started by circulating water at 70°F. The temperature of this water was raised at rates varying from ½°F/hour to 4°F/hour, which resulted in sweating rates from 0.7% to 4.4% per hour. The longest sweating time was 74½ hours and shortest 11½ hours.

In all tests good separation of low setting point and high oil content residue oil was obtained. The cut out from residue oil was fixed at 85°F. because the sweatings at this point were found to contain approximately 70% oil, which as shown in previous experiments, is the point at which no further yield of wax can be obtained by resweating. The residue oil collected varied from 43% to 31%, and the wax from 46% to 49% on charge.

In Tests 57 and 58 the sweated wax was of higher oil content than desired, the wax from Test 58 containing 8.3%. In Test 59 an attempt was made to cut down the time by cooling to 80°F. and starting to sweat at 80°F. This resulted, however, in a decrease in yield to 39.9%, and the wax contained 6.3% oil. It was decided, therefore/

therefore, to repeat Test No.57 (cool to 70°F., start sweating at 70°F. and raise the temperature 3°F/hour), but to sweat a little further to give a wax having about 5% oil. This was done in Test No.60, which showed 40.4% wax of oil content 5.2%. The sweating time was 17 hours, which gives a rate of sweating of 3.5% per hour. The rate is shown graphically in Fig.10.

Test No.60 gave 3.58% residue oil plus single sweat wax on charge per hour (cooling and sweating), which was the highest for all tests giving a sweated wax of the desired oil content.

The Henderson House took 36½ hours to cool and 40 hours to sweat. The residue oil amounted to 37% on charge and the single sweat wax to 47.4% on charge. The wax had an oil content of 4.8%.

Comparison of the experimental sweater under best conditions and the Henderson House is given in Table 28.

Details of all tests are given in Table 29.

TABLE 28/

TABLE 28

	Experimental Sweater	Henderson House
Test No.	60	61
Charge:	1st Strainings	1st Strainings
Setting Point, °F.	103	103
Oil content, %	37.3	37.3
Times:		
Cooling, hours,	3½	36½
Sweating, "	17	40
Total "	20½	76½
Yields: % on charge.		
Residue Oil,	33.0	37.1
2nd Strainings,	26.6	15.5
Single sweat wax,	40.4	47.4
Analyses of Products.		
Residue Oil.		
Setting Point, °F.	80.0	79.0
Oil content, %	78.0	79.0
2nd Strainings.		
Setting Point, °F.	102.0	92.0
Oil content, %	34.0	45.7
Single Sweat Wax.		
Setting Point, °F.	120.9	118.2
Colour, (2" cell)	15.0Y+3.0R	12.0Y+2.3R
Oil content, %	5.2	4.8

(f) Sweating Fifth Strainings.

A few tests were made to determine the optimum conditions for sweating fifth strainings in the experimental sweater, and as before, a comparison with the Henderson House was made.

In all tests the products were residue oil, 6th strainings, and wax of setting point about 115°F. and oil content 0.8%.

The charge had a setting point of 103°F. and contained 17.7% oil, and in every test the residue oil collected had a low setting point and high oil content (62-73%)/

TABLE 29

SWEATING FIRST STRAININGS IN EXPERIMENTAL SWEATER AT DIFFERENT RATES AND IN HENDERSON HOUSE UNDER NORMAL CONDITIONS.

	EXPERIMENTAL SWEATER								HENDERSON HOUSE
Test No:	53	54	55	56	57	58	59	60	61
Charge:	First Strain.	First Strain.	First Strain.	First Strain.	First Strain.	First Strain.	First Strain.	First Strain.	First Strain.
Gallons,	304	293	302	271	299	242	288	284	5620
Temperature, °F.	135	150	150	135	140	140	140	140	150
Setting Point, °F.	103	103	103	103	103	103	103	103	103
Oil content, %	37.3	37.3	37.3	37.3	37.3	37.3	37.3	37.3	37.3
Times:									
Cooling, hours,	3	3	3	3¼	3½	3	2¾	3½	36½
Sweating, "	74½	39	29½	20¾	14	11½	11	17	40
Total "	77½	42	32½	23¾	17½	14½	13¾	20½	76½
Yields of Products: % on chg.									
Residue Oil,	43.4	37.5	37.4	30.6	31.1	34.3	36.0	33.0	37.1
2nd Strainings,	9.9	16.4	15.6	21.0	21.4	16.5	24.1	26.6	15.5
Single sweat wax,	46.7	46.1	47.0	48.4	47.5	49.2	39.9	40.4	47.4
Residue Oil + Single Sw.Wax. % on charge per hour (cooling and sweating)	1.16	1.99	2.59	3.33	4.49	5.76	5.52	3.58	1.10
Conditions:									
Temp.to which charge cooled, °F	70	70	70	70	70	70	80	70	-
Temp.of water at start of sweating, °F.	70	70	70	70	70	70	80	70	-
Temp.of house at start of sweating, °F.	-	-	-	-	-	-	-	-	70
Max.temp.of water during sweating, °F.	108	110	112	112	112	116	113	115	-
Max.temp.of house during sweating, °F.	-	-	-	-	-	-	-	-	118
Rate of increase in temperature, °F/hour	½	1	1 when collect. Residue 2 when collect. Strain.	2	3	4	3	3 to max.	1.2
Sett.Pt. at cut out, °F.									
from Residue,	85	85	85	85	85	85	85	85	85
" 2nd Strainings,	107	107	107	107	107	107	107	110	105
Rate of Sweating. % per hour on charge,	0.7	1.4	1.8	2.5	3.7	4.4	5.5	3.5	1.3
Analyses of Products:									
Residue Oil Sett.Pt., °F.	77.5	79.0	80.0	80.0	79.0	83.0	82.5	80.0	79.0
Oil content, %	86.0	81.1	76.0	77.6	74.6	77.5	74.2	78.0	79.0
2nd Strain. Sett.Pt., °F.	99.5	99.0	99.0	97.0	98.0	102.0	100.0	102.0	92.0
Oil content, %	37.8	43.7	40.8	42.3	43.2	32.1	41.4	34.0	45.7
Single Sw.Wax. Sett.Pt., °F.	119.6	120.6	119.5	120.2	119.2	119.6	120.7	120.9	118.2
Colour (2" cell).	13Y+2.8R	17Y+3.4R	16Y+ 4.2R	15Y+ 4.0R	16Y+ 5.3R	17Y+ 5.2R	15.5Y+ 5.0R	15Y+3.0R	12Y+2.3R
Oil content, %	5.7	4.8	5.7	5.4	7.1	8.3	6.3	5.2	4.8

(62-73%). Details of the tests are given in Table 31.

The products (residue oil plus wax) % on charge per hour was highest (1.20%) in Test No.67, when the charge was cooled to 70°F. and sweating commenced by circulating water at 80°F. The temperature of the water was raised 3°F/hour to a maximum of 112°F. (For rate of sweating see Fig.10). The products % per hour for the Henderson House was 0.50.

Table 30 gives the comparison of the experimental sweater under best conditions and the Henderson House.

TABLE 30

	Experimental Sweater	Henderson House
Test No.	67	68
Charge:	5th Strainings	5th Strainings
Setting Point, °F.	103	103
Oil content, %	17.7	17.7
Times:		
Cooling, hours,	4¼	24½
Sweating, "	26½	61½
Total, "	30¾	86
Yields: % on charge		
Residue Oil,	13.4	16.0
6th Strainings,	63.2	56.7
Wax,	23.4	27.3
Analyses of Wax.		
Setting Point, °F.	114.8	115.0
Colour (2" cell)	3.2Y+0.6R	3.5Y+0.7R
Oil content, %	0.8	0.8

Comparison of Double Sweating and Single Sweating.

During the experiments with hard scale it was proved that double sweating had advantages over single sweating, and should be applied when the yield of single sweat/

TABLE 31

SWEATING FIFTH STRAININGS IN EXPERIMENTAL SWEATER AT DIFFERENT RATES AND IN HENDERSON HOUSE UNDER NORMAL CONDITIONS.

	EXPERIMENTAL SWEATER						HENDERSON HOUSE
Test No:	62	63	64	65	66	67	68
Charge:	5th Strain.	5th Strain.	5th Strain.	5th Strain.	5th Strain.	5th Strain.	5th Strain.
Gallons,	281	292	294	294	292	268	4703
Temperature, °F.	151	150	150	140	141	154	145
Setting Point, °F.	103	103	103	103	103	103	103
Oil content, %	17.7	17.7	17.7	17.7	17.7	17.7	17.7
Times:							
Cooling, hours	4	4½	5¾	4¼	4¼	4¼	24½
Sweating, "	88½	54½	41½	33½	24½	26½	61½
Total, "	92½	59	47¼	37¾	28¾	30¾	86
Yields of Products: % on chg.							
Residue Oil,	16.7	15.1	15.0	15.0	16.0	13.4	16.0
6th Strainings,	53.7	60.6	62.6	62.6	66.2	63.2	56.7
Wax,	29.6	24.3	22.4	22.4	17.8	23.4	27.3
Residue Oil + Wax. (cooling and sweating) % per hour on charge.	0.50	0.67	0.79	0.99	1.18	1.20	0.50
Conditions:							
Temp.to which chg.cooled, °F.	70	70	70	70	70	70	-
Temp.of water at start of sweating, °F.	70	70	70	70	70	80	-
Temp.of house at start of sweating, °F.	-	-	-	-	-	-	77
Max.temp.of water during sweating, °F.	112	112	112	112	115	112	-
Max.temp.of house during sweating, °F.	-	-	-	-	-	-	118
Rate of increase in temp. °F/hour,	½ to max.	1 to max.	2 to max.	3 to max.	3 to max.	3 to max.	0.9 to max
Setting Pt. at cut out,							
Residue,Oil, °F.	85	85	85	85	85	85	85
6th Strainings, °F.	110	110	110	110	111	110	110
Rate of Sweating. % per hr.on charge,	0.8	1.4	1.9	2.3	3.3	3.0	1.2
Analyses of Products:							
Residue Oil Sett.Pt. °F.	75	76½	77	77½	80	76.8	78
Oil content %	73	72.9	71.3	62.2	63.2	63.3	67.6
6th Strainings Sett.Pt. °F.	102	102.5	102.5	103	104.2	103.7	102
Oil content %	15.4	11.9	13.1	9.8	9.2	8.7	11.4
Wax. Sett.Pt. °F.	114	114.8	114.6	115	113.6	114.8	115
Colour (2" cell)	5Y+1.1R	3.5Y+0.5R	3.5Y+0.7R	3.5Y+0.7R	6.2Y+1.0R	3.2Y+0.6R	3.5Y+0.7R
Oil content %	1.0	0.8	0.8	0.8	0.8	0.8	0.8

sweat wax was approximately 50%. Because of the low yield of wax, 23.4%, obtained from 5th strainings, it was considered therefore that this was a case where double sweating should be applied. Tests were made to prove this point and to determine the conditions for double sweating.

The charge had a setting point of 97.5°F. and oil content 42%, and in both tests was cooled to 70°F., and water at 80°F. pumped through the tubes to start the sweating, the temperature being raised at a rate of 3°F/hr. When double sweating, the wax was melted out when the yield was 43% on charge. The wax was cooled and resweated, the maximum temperature of the water being 112°F. The double sweated wax had a setting point of 119°F. and oil content 0.8%, and amounted to 21.2% on original charge. When sweating direct to wax of low oil content, it was necessary to raise the water to 113°F. maximum to keep up the rate of sweating, and in spite of this the wax had a lower setting point than the double sweat product. The wax yield was 20.7% on charge, the setting point being 118.5°F. and oil content 0.9%. The results are summarised in Table 32, and details are shown in Table 33.

The results confirm the previous findings that double sweating is a definite advantage. The time for double sweating was 26.15 hours against 29.5 hours for single sweating. The double sweated wax amounted to 21.2% on charge as compared with 20.7% for the single sweat wax, and/

and the former had a slightly lower oil content.

TABLE 32

		Double Sweating	Single Sweating.
Test No.		69	70
Charge:		5th Strainings	5th Strainings
Setting Point,	°F.	97.5	97.5
Oil content,	%	42.0	42.0
Times:			
Sweating 5th Strainings.			
Charging,	hours,	1	1
Cooling,	"	4½	4½
Sweating,	"	7¾	21
Running out Single Sw.Wax.	hrs.	4	3
		17¼	29½
Sweating Single Sweat Wax.			
Charging,	hours,	1	
Cooling,	"	3	
Sweating,	"	13½	
Running out sweated wax,	hrs.	3	
		20½	
Total cooling,	hours,	7½	4½
" sweating,	"	21¼	21
" time	"	37¾	29½
Yields: % on original charge.			
Residue Oil,		44.7	44.1
6th Strainings,		11.8	35.2
7th Strainings,		22.3	-
Wax,		21.2	20.7
Analyses of Sweated Wax.			
Setting Point,	°F.	119.0	118.5
Oil content,	%	0.8	0.9

The actual time for double sweating would be:-

Sweating 5th Strainings,	17.25 hours
Sweating .435 ton of Single Sweat wax for 20.5 hours,	8.9 "
	26.15 "

(g) Sweating Soft Scale

To complete the experiments on various types of fractions, soft scale was sweated under different conditions/

TABLE 33

COMPARISON OF DOUBLE SWEATING AND SINGLE SWEATING OF FIFTH STRAININGS IN EXPERIMENTAL SWEATER.

Test No.	69		70
	Double Sweating		Single Sweating
Charge	5th Strainings	S.S.Wax from 5th Strainings	5th Strainings
Gallons,	290	271	295
Temperature, °F.	140	142	145
Setting Point, °F.	97.5	113.2	97.5
Oil content, %	42.0	12.0	42.0
Times:			
Cooling, hours,	4½	3	4½
Sweating, "	7¼	13½	21
Total "	12¼	16½	25½
Yields: % on charge			
Residue Oil,	44.7	-	44.1
6th Strainings,	11.8	-	35.2
7th "	-	22.3	-
Wax,	43.5	21.2	20.7
Conditions:			
Temp.to which charge cooled, °F.	70	95	70
Temp.of water at start of sweating, °F.	80	95	80
Max.temp.of water during sweating, °F.	102	112	113
Rate of increase in temp. °F/hour,	3	3 to max.	3 to max.
Setting Point at cut out, °F.	96	110	110
Rate of Sweating, %/hour on charge.	7.2	3.8	3.8
Analyses of Products:			
Residue Oil.			
Setting Point, °F.	79		80
Oil content, %	77.5		80.6
Strainings.			
Setting Point, °F.	91.5	107	104
Oil content, %	49.2	17.2	25.5
Single Sweat Wax.			
Setting Point, °F.	113.2		
Oil content, %	12.0		
Wax.			
Setting Point, °F.		119.0	118.5
Oil content, %		0.8	0.9

conditions in the experimental sweater and in the Henderson House.

The soft scale used had a setting point of 94.5°F. and oil content 35%, and was sweated to give residue oil, 8th strainings and a wax of setting point about 107°F. and oil content about 1.5%. This grade of wax, when heated for four hours at 90°F. under specified conditions, must not show a loss exceeding 5%, and this test was therefore applied to the sweated waxes.

The cut out from residue oil was fixed at 85°F. as in the case of the tests already described. The reason for deciding on 85°F. again was that at this point the strainings had an oil content of 50% and the setting point of the oil free wax in the strainings was 95°F. Wax of setting point about 103°F. is the lowest grade desired in practice and itwas considered that a setting point of 95°F., oil free, was too low to give wax of this setting point.

The sweater was operated at rates ranging from 1.0% per hour to 5.2% per hour, and in every case a residue oil of low setting point and high oil content was obtained. By raising the rate of temperature increase in the circulating water from ½°F/hour to 2°F/hour (Tests 71-73) the total time was reduced from 54½ hours to 20½ hours without any decrease in quality or yield of products.

By/

By raising the water temperature 3°F/hour, however, the wax yield decreased and the loss on heating exceeded the specified maximum. In Test No.76 the temperature was raised 3°F/hour and the total time was 16¾ hours. The wax showed a loss on heating of 6.1% and the yield was 43.1% as compared with 49.3% in Test No.73. It is concluded that the best conditions are those for Test 73, that is cool to 70°F., start sweating with water at 70°F., and raise the temperature 2°F/hour. Under these conditions the residue oil plus wax was 4.21% on charge per hour, as against 0.65% for the Henderson House. The rate of sweating in Test No.73 is shown in Fig.10.

For comparison of the experimental sweater under these conditions and the Henderson House, see Table 34.

TABLE 34

	Experimental Sweater	Henderson House
Test No.	73	77
Charge:	Soft scale	Soft scale
Setting Point, °F.	94.5	94.5
Oil content, %	35.0	35.0
Times:		
Cooling, hours,	3	44
Sweating, "	17½	92
Total "	20½	136
Yields: % on charge,		
Residue Oil,	37.1	39.9
8th strainings,	13.6	11.1
Wax,	49.3	49.0
Analyses of Wax.		
Setting Point, °F.	107.2	107.2
Colour (2" cell)	7.5Y+1.3R	9.0Y+1.8R
Oil content, %	0.9	1.2
Loss on heating 4 hours at 90°F., %	2.4	2.7

Details of all tests are given in Table 35.

(4)/

TABLE 35

SWEATING SOFT SCALE IN EXPERIMENTAL SWEATER AT DIFFERENT RATES, AND COMPARISON WITH HENDERSON HOUSE.

	EXPERIMENTAL SWEATER						HENDERSON HOUSE
Test No:	71	72	73	74	75	76	77
Charge:	Soft Scale	Soft Scale	Soft Scale	Soft Scale	Soft Scale	Soft Scale	Soft Scale
Gallons,	289	294	346	266	296	269	5392
Temperature, °F.	135	148	145	152	144	159	132
Setting Point, °F.	94.5	94.5	94.5	94.5	94.5	94.5	94.5
Oil content, %	35.0	35.0	35.0	35.0	35.0	35.0	35.0
Times:							
Cooling, hours	3	4	3	3¼	3¼	3¾	44
Sweating, "	51½	28½	17½	10½	11	13	92
Total "	54½	32½	20½	13¾	14¼	16¾	136
Yields of Products: % on charge.							
Residue Oil,	36.3	36.1	37.1	41.7	41.8	39.4	39.9
8th Strainings,	15.6	15.3	13.6	12.8	13.2	17.5	11.1
Wax,	48.1	48.1	49.3	45.5	45.0	43.1	49.0
Residue Oil + Wax. % on charge per hour	1.55	2.61	4.21	6.34	6.09	4.93	0.65
Conditions:-							
Temp. to which chg.cooled, °F.	70	70	70	70	70	70	-
Temp.of water at start of sweating, °F.	70	70	70	70	70	70	-
Temp.of house at start of sweating, °F.	-	-	-	-	-	-	62
Max.temp.of water during sweating, °F.	97½	97	103	100	103	106	-
Max.temp.of house during sweating, °F.	-	-	-	-	-	-	97
Rate of increase in temp., °F/hour,	½	1	2	3	3	3	½ to max.
Sett.Pt. at cut out, from Residue, °F.	85	85	85	85	85	85	85
" Strainings, °F.	96	96	98	96	98	100	96.2
Rate of Sweating . % per hour on charge,	1.0	1.8	2.9	5.2	5.0	4.4	0.5
Analyses of Products:							
Residue Oil. Sett.Pt., °F.	75	77	76.5	77.5	77.0	79.5	72.0
Oil content, %	78.1	70.8	66.9	70.2	62.5	57.2	83.5
8th Strainings. Sett.Pt., °F.	89	88	91	88.5	91	94.5	91.5
Oil content, %	43.3	48.8	31.0	41.9	25.0	23.2	35.2
Wax. Sett.Pt., °F.	106.2	106	107.2	106.4	107	107.4	107.2
Colour (2" cell),	7.5Y+1.3R	8.0Y+1.4R	7.5Y+1.3R	9.0Y+1.5R	10Y+2.0R	9Y+1.8R	9Y+1.8R
Oil content, %	1.8	2.7	0.9	2.7	2.1	1.5	1.2
Loss on heating 4 hrs. at 90°F. %	1.9	3.8	2.4	8.0	7.6	6.1	2.7

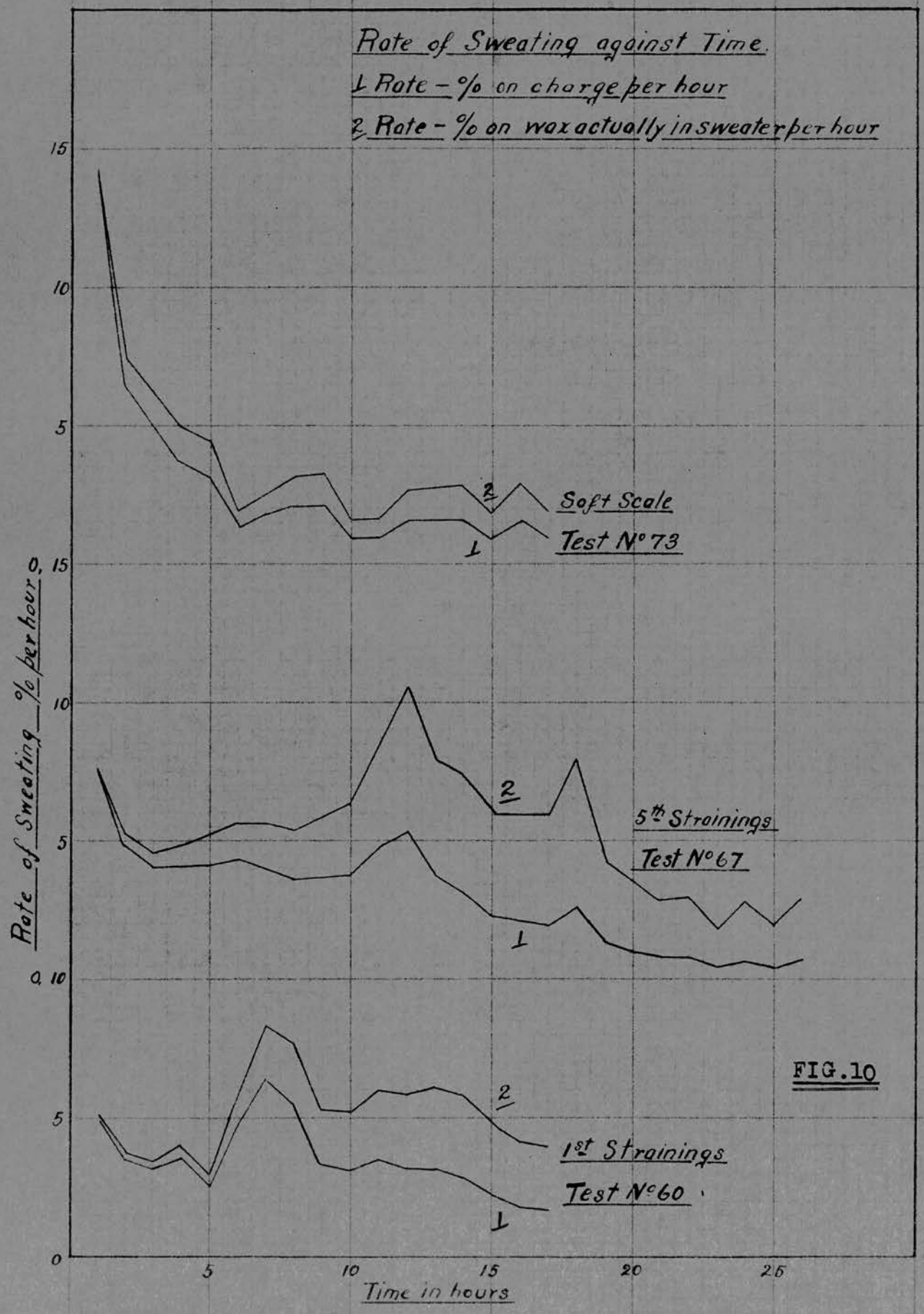

Rate of Sweating against Time.
1 Rate - % on charge per hour
2 Rate - % on wax actually in sweater per hour
Rate of Sweating % per hour
15
10
5
0
2
1
Soft Scale
Test N° 73
0, 15
10
5
0, 10
2
1
5th Strainings
Test N° 67
5
0
2
1
1st Strainings
Test N° 60
5
10
15
20
25
Time in hours

FIG. 10

(4) General Conclusions on Sweating.

The charge to the sweating plant consists of treated hard and soft scales of oil content approximately 15% and 30% respectively. These, by repeated sweating, are converted into waxes ranging in setting point from 130/35°F.-100/05°F., containing from 0.2% to about 1.5% oil and a fraction termed residue oil. Residue oil is transferred to cracking stock because on account of its high oil content resweating would not give a further yield of wax. On an average, residue oil has a setting point of 78°F. and oil content 72%. The sweated waxes amount to 65% on scales charged to the plant, the remainder being the residue oil.

Because of the poor separation of oil and wax in any particular sweating, a large amount of recycling must be done, and to prepare narrow cut waxes low in oil for the minimum of sweating, a proper scheme of recycling must be adhered to. The starting material for any sweating should be such that when the oil removal is complete, the wax left in the sweater has the required setting point and "width of cut". Blending of sweatings should be done on the basis of oil free setting points. In this way fractions containing like setting point waxes are blended for further sweating. The amount of sweating actually carried out can be judged from the fact that for every ton of wax made 4.67 tons of material (original/

original charge plus recycle) are sweated.

The experimental work on semi works scale vertical tube sweaters showed that a vertical tube sweater 17' high and having a cooling and heating surface of 195 sq.ft. per ton of charge could give yields and quality of products very similar to those of the Henderson House, and the time cycle was considerably less. Tests showed that during cooling and sweating the rate of pumping water should be as high as possible to give the lowest possible time of operation. The application of pressure above the wax being sweated was tried out as a means of increasing the rate of sweating, but the results were not favourable.

Detailed experiments were made on the vertical tube sweater (17' high and heating surface 195 sq.ft./hour) to determine the optimum conditions for sweating various fractions, and a direct comparison with the Henderson House made in each case. The comparison between the tube sweater under best conditions and the Henderson House is summarised in Table 36.

In every case the vertical tube sweater gave products of the same quality as the Henderson House. The products percentage on charge per hour (cooling plus sweating) for the experimental sweater was 2.4/6.5 times that of the Henderson House depending on the charge being sweated.

TABLE 36

COMPARISON OF VERTICAL TUBE SWEATER & HENDERSON HOUSE

CHARGE:	Hard Scale		Single Sweat Wax		First Strainings		Fifth Strainings		Soft Scale	
SWEATER:	Vert. Tube	Hend. House	Vert. Tube	Hend. House	Vert. Tube	Hend. House	Vert. Tube	Hend. House	Vert. Tube	Hend. House
Charge.										
Setting Point, °F	120.0	120.0	126.4	126.4	103.0	103.0	103.0	103.0	94.5	94.5
Oil content, %	18.6	18.6	5.0	5.0	37.3	37.3	17.7	17.7	35.0	35.0
Times.										
Cooling, hours,	4	30	2½	25	3½	36½	4¼	24½	3	44
Sweating, "	6½	28	14	67	17	40	26½	61½	17½	92
Total "	10½	58	16½	92	20½	76½	30¾	86	20½	136
Yields. % on charge										
Residue Oil,	-	-	-	-	33.0	37.1	13.4	16.0	37.1	39.9
Strainings,	31.6	31.0	52.7	51.0	26.6	15.5	63.2	56.7	13.6	11.1
Wax,	68.4	69.0	47.3	49.0	40.4	47.4	23.4	27.3	49.3	49.0
Residue Oil + Wax % on charge/hour (cooling & sweating)	6.51	1.2	2.86	0.53	3.58	1.10	1.20	0.50	4.21	0.65
Analyses of Wax.										
Setting Point, °F.	126.3	126.0	131.1	131.4	120.9	118.2	114.8	115.0	107.2	107.2
Oil content, %	6.8	6.4	0.3	0.5	5.2	4.8	0.8	0.8	0.9	1.2

In connection with sweating generally, it was shown that accurate temperature control was of the utmost importance. Time could be wasted by cooling too far and by sweating too slowly at the beginning. On the other hand by raising the maximum temperature a degree or two above the optimum very considerable loss in yield resulted. It is concluded that the only way of knowing whether the proper conditions have been applied or not is to plot rate of sweating (percentage per hour on wax actually in sweater) against time. Sweating rate should be fast at the start and slow towards the finish. A starting rate of 15% per hour was not too fast, but at 30% per hour the saving in time was more than balanced by the loss in yield of products. When preparing single sweat waxes of oil content about 5%, a true rate of sweating towards the finish of about 4% per hour was satisfactory, but when making waxes of low oil content, the rate at the finish should not exceed about 2% per hour.

The gap between setting point of strainings at the cut out and the setting point of the sweated wax varied considerably. For example, when processing hard scale, a cut out setting point of 108°F. gave a wax (7% oil) of setting point 126.3°F., whereas when making wax (0.5% oil) of setting point 131.1°F., it was necessary to have a cut out setting point of 125.5°F.

The/

The question of double sweating as against sweating in one stage was considered in detail and the findings were that double sweating was the proper method, since by this method there was a small reduction in total time, and a slightly greater yield of a slightly better wax was obtained. For the minimum total time by double sweating, the first sweating should be stopped when the yield of wax is approximately 50% on charge.

The importance of maintaining the oil content of the scales charged to the sweaters as low as possible was demonstrated by tests in which scales of various oil contents were processed. Increase in oil content of scale resulted in longer time of sweating, lower yield of product, and raised the setting point of the wax. The graph showing relationship between oil in scale and yield of wax proves that at 70% oil in charge no wax can be separated by sweating. The total yield of wax (that is by direct sweating and by sweating strainings completely to residue oil and wax) which can be obtained from any scale has been shown to be about 2.8 times the yield which can be realised by direct sweating.

A survey of technical literature shows that while the theory of sweating has been discussed [24,25], and many descriptions of plants given, very little experimental work of practical importance has been published, and in cases/

cases where details of actual plant operation have been given important facts such as oil content of charge and of products are lacking.

Pyhälä[26] recommended rapid increase in temperature at the start of sweating because he considered that to keep the oil in contact with the wax longer than necessary resulted in low yield of sweated product. Although from the experiments with shale waxes it was concluded that fast sweating at the beginning was advisable, in no case was it found that slow sweating gave lower yield. The faster sweating tended to give lower yields, but the products per hour were increased.

Allan[27], one of the designers of the Alanmor stove, giving details of operating conditions on this plant, indicated that the charge was cooled to the point where 5/6% was left in the liquid state and the temperature of heating water then controlled to be very close to the setting point of the wax in the stove. The tests with the vertical tube sweater on shale waxes showed that the water temperature was considerably below the setting point of the wax, and that increasing the maximum temperature seriously decreased the yield. When sweating hard scale to wax of setting point 126°F. the maximum temperature of the water was only 114°F. and when making wax of setting point 131°F. the water maximum temperature was 127°F. In the/

the case of first strainings, the wax had a setting point of 121°F. and the water maximum 115°F. When sweating 5th strainings, the maximum temperature was 112°F. to give a product of setting point 115°F., while when making wax of setting point 107°F. from soft scale, the maximum temperature was 103°F. Taking the figures recorded by Allan for plant runs, it has been calculated that when making yellow wax, the rate of sweating varied from 4-16% on charge per hour, with an average of 8% per hour. When sweating to white wax the rate varied from 2.5% to 6%, the average being 3% per hour. By calculating the rates when collecting the first fraction and the second fraction in each sweating, there does not appear to have been any attempt made at sweating fast at the start of the process. The wax yield for all sweatings varied from 30% to 50%on charge.

Donnell and Burch[28], describing tests with an experimental pan sweater, concluded that the yield varied with the sweating time, the slower the sweating the greater the yield. They concluded that if the oil content of a charge was above 50% no wax could be obtained by sweating because it was difficult, if not impossible, to obtain a sweatable crystal structure. From their own results, however, there is no evidence that the low yield is due to anything but the high percentage of oil, and this/

this is confirmed by the tests made on shale waxes. When sweating scales of various oil contents, the relationship between yield and oil in charge was found to be linear.

DECOLORISATION OF UNFINISHED WAXES.

The waxes (unfinished waxes) from the sweating plant are high in colour and must be decolorised before being moulded into cakes. Various bleaching agents have been tried at different times, but the best results are obtained by percolation through floridin, a form of Fuller's Earth found in Florida, U.S.A.

A description of the refinery method of carrying out this treatment and the results of laboratory experimental work to determine the conditions necessary to give maximum efficiency are given.

(1) Filtration through Granular Floridin.

The wax to be decolorised is settled till free from moisture and then filtered through previously ignited Floridin of 16-30 mesh per inch. The Floridin is contained in vertical jacketed filters about 14 ft. high, the wax to be decolorised passing down through the column at the rate of approximately 0.5 ton of wax per hour per ton of Floridin. The wax leaving the filter is low in colour at the start of a filtration and gradually increases. See Table 37. When the filtered wax is at the desired colour for the particular grade the filtration is stopped, the Floridin washed with naphtha to remove wax, and then steamed to remove naphtha. The mixture of naphtha and wax is separated by steam distillation, the naphtha being used/

used again and the wax returned for resweating. As already mentioned (page 48) the quantity of wax returned amounts to about 20% of the wax charged. After steaming, the Floridin is re-activated by igniting in a rotary furnace in presence of excess air after which it is ready for further use.

Floridin can be used indefinitely in this way, but sufficient fresh Floridin must be added regularly to make up for mechanical loss. The quantity of fresh Floridin required is about 2% of the Floridin actuall used, and is equivalent to 0.013 ton per ton of wax decolorised. Table 38 gives an idea of the efficiency of fresh Floridin and Floridin after being recovered up to 13 times. When decolorising unfinished 125/30 wax to a colour of 1.5Y (18" cell) by means of ignited fresh Floridin, 6 gms. of wax per gm. of Floridin could be obtained, while with Floridin which had been recovered 13 times, the quantity of wax at the colour was 2 gm. per gm. of Floridin.

Table 39 shows the quantity of fresh Floridin necessary to decolorise various grades of wax to specification colour. As well as removing colour, Floridin treatment decreases the oil content of wax. To illustrate this point, the oil contents of different waxes before and after treatment are also given in Table 39.

TABLE 37/

TABLE 37

Grade and colour of unfinished wax,	P 5/30 10.0Y+2.6R (6" cell)	106/08 7.0Y+1.3R (2" cell)
Treatment.	**Colour of treated wax 18" cell**	**Colour of treated wax 18" cell.**
1 gm.Wax/gm.Floridin,	0.4Y	1.0Y
2 " " " "	0.7Y	1.5Y+0.1R
3 " " " "	0.8Y	2.5Y+0.1R
4 " " " "	0.9Y+0.05R	4.5Y+0.2R
5 " " " "	1.2Y+0.05R	6.0Y+0.25R
6 " " " "	1.5Y+0.05R	9.0Y+0.3R
7 " " " "	-	11.0Y+0.6R
8 " " " "	-	12.5Y+0.6R
9 " " " "	-	14.0Y+0.9R

TABLE 38

DECOLORISATION OF UNFINISHED 125/30 WAX USING FRESH FLORIDIN AND FLORIDIN RECOVERED UP TO THIRTEEN TIMES.

No. of times Floridin recovered	Colour of treated wax (18" cell)	Gms.of wax per gm. of Floridin
Fresh Floridin	1.5Y+0.05R	6.0
1	1.5Y+0.1R	4.5
2	1.5Y+0.05R	4.75
3	1.5Y+0.05R	4.0
4	1.5Y+0.05R	3.25
5	1.5Y+0.05R	3.25
6	1.5Y+0.05R	4.0
7	1.5Y+0.05R	4.25
8	1.5Y+0.05R	3.5
9	1.5Y+0.05R	3.25
10	1.5Y+0.1R	1.75
11	1.5Y+0.1R	2.5
12	1.5Y+0.1R	2.75
13	1.5Y+0.05R	2.0

—

TABLE 39

DECOLORISATION OF DIFFERENT GRADES OF WAX TO SPECIFICATION COLOUR USING IGNITED FRESH FLORIDIN.

Grade of Unfinished wax,	125/30	122/25	118/20	110/15	106/08
Colour of unfinished wax,	10.0Y+ 2.6R 6"cell.	8.0Y+ 1.8R 6"cell	20.0Y+ 2.8R 6"cell	9.0Y+ 1.8R 2"cell	7.0Y+ 1.3R 2"cell
Colour of finished wax,	1.5Y+ 0.05R 18"cell	2.0Y+ 0.1R 18"cell	3.5Y+ 0.2R 18"cell	14.0Y+ 1.1R 18"cell	14.0Y+ 0.9R 18"cell
Gms.of finished wax obtained per gm. of floridin,	6.0	5.75	5.0	8.0	9.0
Oil content of unfinished wax, %	1.05	0.9	0.95	2.6	2.6
Oil content of finished wax, %	0.8	0.5	0.9	2.0	2.0

(2) Experiments to determine best conditions of Operation.

The theory ofdecolorisation of oils by means of Fuller's Earth, Bauxite and other such materials, has been widely studied, and it has been established that the adsorption follows Freundlich's law. This subject is dealt with in text books by Gurwitsch[29] and by Kalichevsky and Stagner[30], while experimental proof of the Freundlich law as applied to adsorption of sulphur and colour compounds by means of Bauxite was produced by Dunstan, Thole and Remfry[31]. Rodgers, Grimm and Lemmon[32] proved the application of the equation to the decolorisation of a number of oils by several adsorbents.

Gurwitsch[29] considered that polymerisation occurred during Floridin treatment because by extracting the colouring matter from Floridin which had been used, and adding the extract to the decolorised oil the resultant mixture was of higher colour than the original oil. Rideal and Thomas[33] found when comparing different varieties of Fuller's earth that as well as adsorption, which depended on the specific surface, catalytic activity was also present, and came to the conclusion that the iron content of the Fuller's earth may have been the governing factor in this catalysis.

In spite of the fact that the decolorising

of/

of oils by different materials follows a general law, it is always emphasised that the best conditions for any treatment must be determined by experiment. Tests were therefore made to find the effect of certain variables on the decolorising of waxes by means of Floridin.

The treatments were effected by running molten wax through Floridin which was contained in a small vertical filter similar in design to the large plant. The temperature of treatment was controlled by pumping water of required temperature through the jacket of the filter.

Rate of Filtration.

Unfinished 118/20 wax was filtered through recovered Floridin at different rates. The same quantity of wax was collected in each case and the colours determined. The results are given in Table 40.

TABLE 40

Unfinished Wax.		Rate of filtration. gms.wax/ gm.of Floridin/ hour.	Quantity of wax filtered, gm.wax/ gm.of Floridin	Temp.of filtration. °F.	Colour of treated wax 6" cell.
Grade	Colour 6" cell				
118/20	13.5Y+3.0R	2.0	3	160	3.0Y+0.25R
118/20	do.	1.0	3	160	3.0Y+0.15R
118/20	do.	0.5	3	160	2.7Y+0.2R

This would indicate that within the limits investigated the rate has not much effect on the colour.

The/

The slower the rate, however, the better the colour.

Temperature of Treatment.

Different grades of unfinished wax were filtered through recovered Floridin at different temperatures to find if temperature had any influence on the efficiency of treatment. 3 gms. of wax per gm. of Floridin were collected in each test, and the rate of filtration was 1 gm. of wax per gm. of Floridin per hour. Table 41 shows the results.

TABLE 41

Grade and colour of unfinished wax	Unfinished Wax 106/8 20.0Y+7.8R 6" cell	Unfinished Wax 118/20 13.5Y+3.0R 6" cell	Unfinished Wax 125/30 7.5Y+1.9R 6" cell
Temperature of treatment	Colour of treated wax 6" cell	Colour of treated wax 6" cell	Colour of treated wax 18" cell
10°F. above S.P. of wax,	8.5Y+1.05R	2.2Y+0.1R	5.2Y+0.3R
40°F. do.	10.0Y+1.35R	3.0Y+0.15R	6.0Y+0.4R
70°F. do.	11.0Y+1.55R	4.0Y+0.4R	7.0Y+0.65R

In each test the lower the temperature of treatment the lower the colour of the filtered wax.

To find if this improvement was due to the effect of heat on wax, a sample of unfinished 118/20 and a sample of finished 118/20 were heated to different temperatures and the colours determined.

TABLE 42/

TABLE 42

	Unfinished 118/20 Wax	Finished 118/20 Wax.
Original colour, 6" cell,	15.0Y+2.7R	2.2Y+0.1R
Colour after 5 hours at 130°F.	13.0Y+3.0R	2.2Y+0.1R
" " 5 hours at 160°F.	13.0Y+3.0R	2.2Y+0.1R
" " 5 hours at 190°F.	15.0Y+3.1R	2.2Y+0.1R

The increase in colour at the higher temperatures is therefore not due to the effect of heat on the wax, and the better decolorisation at the lower temperature must be due to the fact that the Floridin treatment is more effective the lower the temperature.

Rauch[34], when giving points to be remembered when decolorising oils by filtration through Fuller's earth, states that the higher the temperature the faster the flow, and the poorer the bleach; also the higher the temperature the less viscous the oil, the more intimate the contact and the better the bleach. These points do not agree with the results given above when decolorising shale waxes. The rate of flow was the same throughout the tests, and therefore the high colour at high temperature was not due to the rate of filtration, and in any case the first experiments showed that the rate did not have much effedt on the colour. Kalichevsky and Stagner[39] state that the temperature is important and must be determined experimentally for each stock. These authors/

authors conclude that waxes should be decolorised at a temperature about 25°F. above the setting point, and that too high a temperature should be avoided.

Recycling.

In the refinery when any unfinished wax has been decolorised to the specification limit, the Floridin is still capable of removing colour. Tests have been made to find what benefit would be derived if the filtration was continued and a second fraction, above specification colour, collected for refiltering. Unfinished 125/30, 122/25 and 118/20 waxes were decolorised by means of recovered Floridin, 2 gms. of wax of specification colour per gm. of Floridin being collected. In each case the filtration was continued to give a further 2 gms. of wax per gm. of Floridin. This second fraction was then compared with the original unfinished wax as regards colour and colour after filtration through recovered Floridin. In order to give comparative results the temperature and rate of filtration and the quantity of wax collected were the same throughout the tests, the results of which are summarised in Table 43.

In each test the fraction for refiltering had a colour above specification, but much lower than that of the original unfinished wax, and when decolorised gave a product of slightly lower colour than was obtained from the original unfinished wax. The slight improvement in colour/

colour when the wax had been filtered twice is at first sight surprising considering the low colour of the charge to the second filtration, but this point has been long recognised and follows from the fact that the decolorisation obeys Freundlich's law of adsorption. There is, however, a small but definite advantage to be gained by filtering all unfinished wax through floridin which has been used, but not recovered, before decolorising to specification colour by means of recovered Floridin.

TABLE 43

Wax	Colour	Colour after treatment - 2 gms.of wax/gm. of Floridin
Unfinished 12 5/30, 2 gms.wax/gm. Floridin above spec.colour for refiltering.	6.0Y+1.6R(6"cell)	1.5Y+0.1R (18" cell)
	5.5Y+0.6R(18" cell)	1.2Y+0.1R (18"cell)
Unfinished 122/25, 2 gms.wax/gm. Floridin above spec.colour for refiltering.	6.5Y+1.6R(6"cell)	2.0Y+0.15R(18"cell)
	6.5Y+0.8R(18"cell)	1.6Y+0.15R(18"cell)
Unfinished 118/20 2 gms.wax/gm. Floridin above spec.colour for refiltering	8.0Y+2.0R(6"cell)	3.5Y+0.4R (18"cell)
	9.0Y+1.1R(18"cell)	3.0Y+0.3R (18"cell)

Activation of Floridin.

Floridin before activation contains a large percentage of water and is a very poor decolorising agent. Various opinions have been expressed as to how much of this/

this water should be removed to give a decolorising agent of maximum efficiency. Gurwitsch[29] says that partial drying of Floridin increases its decolorising power and that this attains a maximum at a certain water content (about 11%) and then decreases with further drying until with completely dried Floridin the decolorising power is almost lost. Kauffman[35] found the water content to be a variable factor. Some earths decolorised equally well before and after removal while others lost some of their bleaching power when moisture was driven off. Kalichevsky and Stagner[30] state that some Fuller's earths are ignited at 500-900°F in order to expel part or nearly all of the water which they contain, while Funsten[36] recommends heating fresh earth to a temperature of 900°F. for 15 minutes, resulting in the dried material containing about 4% volatile matter. When describing Floridin treatment of shale waxes, Bailey[13] says that all moisture, free and combined, must be driven off, and recommends a temperature of 700-900°F.

A sample of fresh Floridin was found to contain 20% water, and after heating at 1000°F. this was reduced to 6%. Unfinished 125/30 wax, when treated with Floridin (6 gms. of wax per gm. of Floridin) activated under these conditions, gave a colour of 1.5Y+0.05R (18" cell), while under the same conditions Floridin, previous/

previous to activation, gave a product having a colour of 4.0Y+0.7R (6" cell).

A number of tests have been made to find the best temperature for reactivation of used Floridin. The used Floridin, which contained 17% volatile matter, was heated in a muffle for 20 minutes at different temperatures, and the activated Floridin was evaluated by decolorising unfinished 118/20 wax, 3 gms. of wax per gm. of Floridin being collected. The temperature of treatment and rate of filtration were kept constant throughout the tests. The volatile matter in the Floridin samples was determined by heating a finely ground portion for one hour at a temperature of 750°C. The results are shown in Table 44.

TABLE 44

Temperature of activation, °F.	Wax Colours		Volatile matter in activated Floridin %
	Unfinished Wax 6" cell	Decolorised Wax, 3 gms./gm. of activated Floridin. 18" cell	
1400	14.0Y+2.4R	7.2Y+0.65R	2.16
1200	do.	5.0Y+0.5R	3.55
1100	do.	3.5Y+0.15R	3.90
1000	do.	3.5Y+0.1R	5.00
900	do.	4.2Y+0.35R	6.34
800	do.	6.7Y+1.0R	7.50
600	do.	12.0Y+1.2R	10.25

This shows that the filtered wax of lowest colour was obtained from Floridin which had been ignited at/

at 1000°F., resulting in the Floridin containing 5.0% of volatile matter. The Floridin ignited at 1100°F. contained 3.9% volatile matter and the decolorised wax was nearly as good as from the Floridin activated at 1000°F. With a time in the muffle of 20 minutes, 1200°F. was definitely too high, and 800°F. definitely too low.

Tests were then carried out to find the effect of time of heating during activation. Used Floridin was heated at 1000°F. and at 800°F. for various periods and the activated Floridin tested as before.

TABLE 45

Temperature of activation. °F.	Time at the temperature. Minutes.	Wax Colours		Volatile Matter in activated Floridin %
		Unfin. Wax. 6" cell	Decolorised wax. 3 gms./gm. of activated Floridin. 18" cell.	
1000	10	11.0Y+2.5R	6.0Y+0.9R	8.38
1000	20	do.	5.0Y+0.6R	6.07
1000	40	do.	4.5Y+0.6R	4.30
1000	80	do.	6.0Y+0.9R	3.83
1000	160	do.	5.6Y+0.6R	1.97
800	10	do.	8.5Y+1.3R	8.55
800	20	do.	6.5Y+0.7R	6.87
800	40	do.	5.0Y+0.55R	6.27
800	80	do.	4.8Y+0.5R	4.50
800	160	do.	5.5Y+0.6R	3.48

When the temperature was 1000°F. a time of heating of 40 minutes gave the best activated Floridin, the volatile matter being 4.3%. With a temperature of 800°F. the best time of heating was 80 minutes, and the volatile matter/

matter in this case was 4.5%. The Floridin heated for 40 minutes at 1000°F. was very similar in decolorising power to Floridin heated for 80 minutes at 800°F., and it is concluded that reactivated Floridin should contain about 5% volatile matter. In the refinery the Floridin takes 20 minutes to pass through the rotary furnace, and therefore the temperature of ignition should be about 1000°F.

The relationships between temperature of activation and volatile matter, and between time at the temperature and volatile matter in activated Floridin, are shown graphically in Fig.11.

In the literature various temperatures are quoted for the reactivation of used Floridin, but most authors recommend 1000-1100°F. According to Funsten[36] reactivation should take place at 1100-1200°F. for 15 minutes, and the resultant material should contain not less than 2% volatile matter.

Conclusions.

Within the limits investigated - 0.5 gm./2.0 gm. of wax per gm. of Floridin per hour - and with the filter column used, the rate of filtration had no appreciable influence on the colour of the treated wax. The best colour, however, was obtained at the lowest rate, and the highest colour at the fastest rate.

To obtain maximum decolorisation, the temperature during filtration should be as low as possible.

Better/

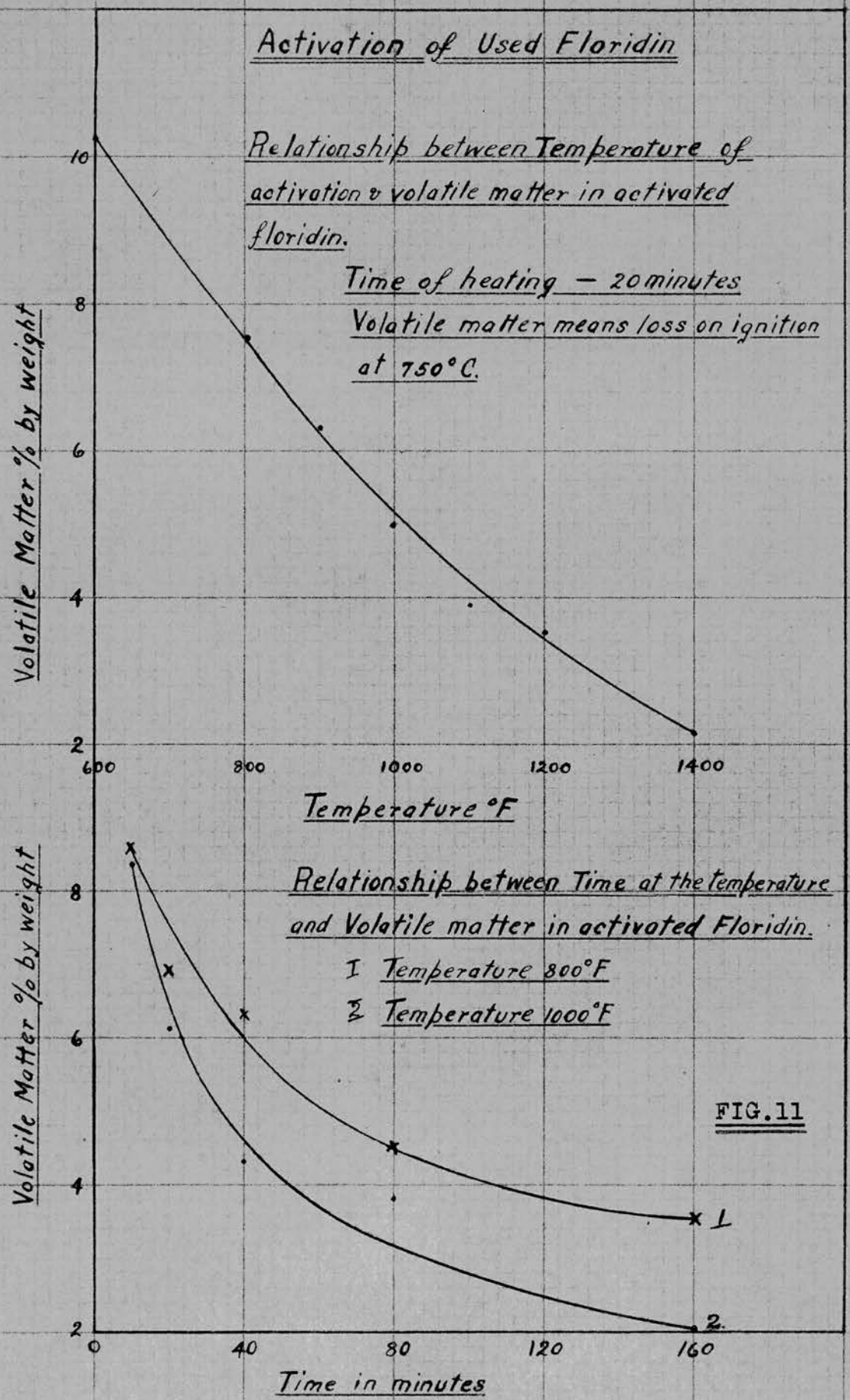

Activation of Used Floridin
Relationship between Temperature of activation & volatile matter in activated floridin.
Time of heating — 20 minutes
Volatile matter means loss on ignition at 750°C.
Volatile Matter % by weight
10
8
6
4
2
600
800
1000
1200
1400
Temperature °F
Relationship between Time at the temperature and Volatile matter in activated Floridin.
I Temperature 800°F
2 Temperature 1000°F
Volatile Matter % by weight
8
6
4
2
0
40
80
120
160
1
2.
Time in minutes

FIG.11

Better efficiency of decolorisation resulted from filtering all unfinished wax through Floridin which had been used but not recovered before finally decolorising to specification colour by means of recovered Floridin.

For a time in the furnace of 20 minutes used Floridin should be activated by heating to a temperature of 1000°F. The best recovered Floridin contained about 5% volatile matter.

CASTING OF WAXES

(1) Pan Moulding.

In the shale industry, the melted wax, after being decolorised, is run into trays and allowed to cool to atmospheric temperature. These trays are 24" x 12" x 2½", and give a cake weighing about 14 lbs. This method of allowing trays of wax to cool under atmospheric conditions is known as pan moulding and gives a cake smooth on the bottom but rough on the surface in contact with the atmosphere. If the temperature of casting is only a few degrees above the setting point of the wax the roughness of the top surface may be objectionable, but by charging the pans with wax at a higher temperature and by preventing draughts, a satisfactory finished cake can be prepared. The pans can be emptied about 18 hours after charging.

(2) Press Moulding.

In countries where the atmospheric temperature is high, pan moulding has been replaced by press moulding. The procedure in this case is to cool the molten wax in moulds of same size as the trays described above, by pumping cold water through cells which form the moulds. The cakes of wax made in this way are of more uniform appearance since all surfaces are smooth. Press moulded waxes in general have a more opaque appearance than pan moulded.

Shale/

Shale waxes of all grades have been cast in an experimental press mould and in all cases a more opaque wax was obtained than by the normal pan moulding method. The press moulded wax is generally considered to be of more attractive appearance.

(3) Mottling.

Although all grades of shale wax are more or less transpareent, occasionally cakes have been seen which show mottles (these are opaque patches) in an otherwise transparent cake. In extreme cases the cakes may be split horizontally at the centre, the top surface of the cake being slightly raised.

Various experiments were made to find the cause of mottling.

Examination of Mottled Cakes.

A cake of wax which showed three distinct layers and was split at the centre, was cut horizontally into three sections: (1) top, which was transparent, (2) middle, which was opaque, and (3) bottom, which was similar in appearance to (1).

Each section was melted, poured into a metal dish and allowed to cool at atmospheric temperature. Examination of these samples and of an average sample of the original cake showed:-

TABLE 46/

TABLE 46

	Setting Pt.	Oil Content	Appearance
Original sample,	127.6°F.	1.5%	Badly mottled
Top section,	127.4°F.	1.55%	do.
Middle section,	128.4°F.	1.25%	do.
Bottom section,	127.5°F.	1.7%	do.

This shows that the middle portion had a higher setting point than top or bottom, and was of lower oil content. This lower oil content at the centre was surprising, but similar results were obtained when the tests were repeated. The centre portion, when re-cast, was not any more mottled than the top or bottom.

Effect of casting at different Temperatures.

A wax of setting point 128.8°F. and oil content 1.5% when cast and cooled under different conditions, gave the results shown in Table 47.

TABLE 47

Conditions of Casting.	Appearance of Cake
(1) Melted wax heated to 200°F. and cooled at atmospheric temp. (normal conditions)	Badly mottled
(2) Heated to 140°F. and cooled at atmospheric temperature	Badly mottled
(3) Heated to 140°F. and cooled slowly.	Badly mottled
(4) Heated to 140°F. and cooled quickly	Mottling greatly reduced and wax more opaque
(5) Heated to 250°F. and cooled at atmospheric temperature.	Badly mottled.
(6) Heated to 250°F. and cooled slowly.	Mottling reduced and wax more transparent.
(7) Heated to 250°F. and cooled quickly.	Mottling greatly reduced and wax more opaque.

Tests/

Tests (4) and (7), i.e. quickly cooled by means of iced water, gave a more opaque cake with mottling greatly reduced.

This is in agreement with previous tests comparing pan moulding and press moulding. Heating to 250°F. and cooling slowly by placing in an electric bath, the temperature of which was gradually lowered, reduced the mottling and gave a more transparent wax (Test (6)).

Effect of Oil.

Different amounts of oil were added to a wax which when cast normally showed no mottling. The wax used had a setting point of 131.5°F. and oil content 0.25%.

TABLE 48

	Oil content of Wax.	Appearance after normal casting (heated to 200°F. and cooled at atmospheric temperature)
(1)	0.25% (original wax)	No mottling; transparent.
(2)	0.5%	do. do.
(3)	0.75%	Badly mottled; transparent.
(4)	1.0%	Very badly mottled; opaque.
(5)	1.25%	Worse than (4); very opaque and mottles difficult to see.
(6)	1.75%	Worse than (5)
(7)	2.25%	Worse than (6)

The addition of oil to a wax which did not show mottling has induced mottling when the total oil in the wax was 0.75% or higher. With oil of 1.0% the cake was very badly mottled and opaque.

A /

A wax, which when cast gave a badly mottled cake, was sweated in an experimental sweater, 10% strainings being removed. The sweated wax, when cast, gave a cake free from mottling, while the strainings when cast gave a badly mottled cake.

Effect of "Width of Cut".

It was thought that the splitting of a cake due to excessive mottling might be due to the wax being brittle and less plastic than usual, and to find if there was anything in this, 10% of 115/18 wax was added to a wax of setting point 128°F. which showed mottling and splitting of the cake. The cakes from this blended wax were very similar to the original. The test was repeated using 10% of 106/8 wax in place of the 115/18 wax, and the same result was obtained, and so the presence of plastic wax does not prohibit mottling.

Effect of Air.

An apparatus was fitted up so that wax could be kept liquid under a pressure of about 30 mms. mercury, and then allowed to solidify under the reduced pressure.

A wax, of setting point 126.2°F. and oil content 2%, which when cast and cooled in the normal manner gave a badly mottled cake, was melted and kept liquid in the above apparatus under a pressure of 30 mms. mercury for one hour, and then allowed to solidify still under/

under the 30 mms. mercury pressure. When the reduced pressure was applied to the liquid wax, bubbles of gas could be seen coming to the surface. The cake of wax obtained showed no mottling and was transparent. The setting point, oil content and weight of the cake were the same as those of the original wax, and therefore oil had not been removed by casting under reduced pressure.

Another experiment in which the wax was kept liquid for one hour under the reduced pressure and then allowed to solidify at atmospheric pressure gave a cake which was badly mottled.

Vacuum cast transparent wax, free from mottling, when melted and cast in the usual way gave a badly mottled cake.

2% of oil was added to wax which had previously been shown to give a badly mottled cake when cast normally. The high oil content wax (4%) was cast and cooled under reduced pressure and in the normal way. The cake from reduced pressure casting was transparent and free from mottling, while the normal method of casting gave a cake badly mottled and split at the centre.

It has already been shown that wax cooled quickly at atmospheric pressure was more opaque than wax cooled in the normal way, and a test was made to find what would be the effect of cooling the wax quickly under reduced/

reduced pressure. A wax was heated for one hour at a pressure of 30 mm. mercury and then quickly cooled still under the low pressure by means of iced water. For comparison the same wax was cooled slowly under reduced pressure. The cake from the quick cooling was not quite so transparent as the wax obtained by the slower cooling, but was not opaque.

Finally attempts were made at inducing mottling in a wax, which, cast normally, showed no mottling. The wax, setting point 135.9°F. and oil content nil, was melted, cooled, and allowed to solidify under a pressure of 35 lbs. per sq.in. gauge. The resultant cake was badly mottled, and the cake split at the centre.

Several of the above tests on effect of air were repeated using different grades of wax, and the same results were obtained.

The tests show that waxes high in oil gave transparent cakes free from mottling when dissolved air was removed and the wax allowed to solidify under reduced pressure. By removing dissolved air and allowing to solidify at atmospheric pressure a mottled cake was obtained, and therefore the air must be drawn in during the solidification. It has also been proved that wax of very low oil content can give mottled cakes if the melted wax is made to solidify under increased pressure.

Conclusions/

Conclusions.

As a result of the tests made to find the reason for opaqueness and mottling in cakes of wax, it has been shown that:-

(1) Quick cooling as in press moulding gave a wax with less mottling but more opaque than slow cooling (pan moulding).

(2) With pan moulded wax the addition of oil to a wax-free from mottling induced mottling if the oil content was 0.75% or above. The higher the oil in the wax the more opaque and mottled was the cake.

(3) The addition of low setting point plastic wax did not prevent mottling.

(4) Wax of high oil content, when cast under reduced pressure and cooled at atmospheric temperature, gave cakes which were transparent and free from mottling.

(5) Wax kept liquid under reduced pressure long enough to remove dissolved air, when allowed to solidify at atmospheric pressure and temperature gave a mottled cake. This would indicate that air is drawn into the wax during solidification.

(6) Casting of oil-free wax under increased pressure and atmospheric temperature resulted in a mottled cake.

(7) The cause of mottling in wax is the air which enters during cooling and solidification, but the phenomenon is influenced by the oil content of the wax. In normal pan moulded wax excessive opaqueness and mottling are evident only if the oil in the wax is high.

Campbell and Wilson[21], reviewing the early literature on mottling, show that there was difference of opinion as to the cause. One author found that the presence of not less than 0.5% oil gave an opaque wax, while another/

another author stated that a transparent wax containing 3.6% oil had been prepared. It was also stated that transparency only occurred in a wax composed of constituents not differing widely in setting point.

Carpenter[37] states that mottling is bound up with the presence of air, and that the addition of anti-mottling compounds gives an opaque wax due to the even separation of air in minute quantities throughout the mass instead of in patches. Carpenter found that mottling was increased by the presence of oil.

Katz[38] examined mottles under the microscope and describes the appearance as being typical of a "blow hole" in a metallic casting, and says that they probably arise in the same way.

Mottling is a drawback in candle making, and Allan[39], when describing methods which have been used in overcoming the trouble, mentions steaming to remove dissolved air. This, however, would not appear to be a satisfactory method, as in the tests described on page 122 the removal of dissolved air by application of reduced pressure only gave a wax free from mottling if the low pressure was maintained till the wax had solidified.

Recently Scott-Harley[40] and Lord[41], in connection with work on transition points, to which reference will be made later, observed wax cooling under the microscope. The former noted that below the setting point/

point a contraction in volume took place and air entered the wax. At a still lower temperature with certain waxes a transition from one crystal form to another took place, and this was accompanied by the release of air. Scott-Harley also says that it is notable that waxes which are definitely translucent do not show a transition point, while waxes which are decidedly opaque generally show a marked transition point.

Lord found that when wax was melted and allowed to solidify and cool slowly under the microscope segregation of air into the crystal boundaries could be seen to take place during the transition from one type of crystal to another. He concluded that mottling is intimately connected with the presence of air, increases with rising oil content, and does not take place in absence of air even if considerable oil is present.

Lord's observations on the cooling of waxes are very similar to those of Scott-Harley, and his conclusions are in agreement with those arrived at from the experiments on casting shale waxes under different conditions.

CHEMICAL & PHYSICAL PROPERTIES OF WAXES.

(1) Composition.

A considerable amount of work has been published on the hydrocarbons present in waxes, and these have been proved to belong mainly to the normal and iso-paraffin series with the general formula $C_nH_{2n} + 2$. Buchler and Graves[42] found hydrocarbons ranging from $C_{18}H_{38}$ (S.P. 80.5°F.) to $C_{43}H_{88}$ (S.P. 182°F.) in American petroleum, while Carpenter[37], by repeated crystallisation from solvents separated hydrocarbons ranging from $C_{21}H_{44}$ (S.P.106°F) to $C_{57}H_{116}$ (S.P. 206°F.) from Burmah crude oil. The hydrocarbons above $C_{34}H_{70}$ were present only in small quantities and were deposited in the pipe line at the oil wells, and Carpenter concludes that in the crude as received at the refinery most, if not all, of the hydro-carbons from $C_{21}H_{44}$ up to $C_{34}H_{70}$ are present. The chief constituents, which appeared to be better defined than the others, had setting points of 106°F., 111-113°F., 117-118°F., 126-127°F., 135-136°F., 142-145°F., 151-153°F., and a definite though small quantity with setting point of 160-162°F.

In the higher setting point fractions Carpenter found that fractions of the same setting point had different molecular weights, and in other cases fractions with the same molecular weights had different setting/

setting points. From this he considered it possible that isomers of the normal paraffin or saturated naphthenes might be present.

Carpenter also studied the properties of waxes from Assam, Dutch East Indies and America, and states that these gave results very similar to Burmah crude.

Professor Francis[43] of Bristol University and assistants made a study over a number of years of wax (setting point 131/133°F) from Scotch shale oil. By repeated distillation under high vacuum seven fractions of constant boiling point were separated, and these fractions amounted to about 80% of the original wax. Air oxidation at 100°C. of the seven fractions was carried out, and the unoxidised material left after a certain time was found to be exactly similar to the original material, from which it was concluded that the seven constant boiling point fractions were very probably pure hydrocarbons. Piper, Brown and Dyment[44] made an X-ray examination of the seven constant boiling fractions and found that these belonged to a homologous series and deduced the number of carbon atoms. For comparison, three synthetic normal hydrocarbons were photographed. These photographs and those of the seven fractions were compared with results previously obtained by Müller and Saville[45] for synthetic normal hydrocarbons and/

and found to be identical. There was no evidence of the presence of iso-paraffins.

Francis and Wood[46] determined the boiling points of the seven fractions at different pressures and compared these with results for synthetic aliphatic normal hydrocarbons, and decided that the seven hydrocarbons isolated from shale wax were as given in Table 49:-

TABLE 49

	Molecular Formula	Boil.Pt. at 15 mm. Hg Press. °C.	Melting Point. °F.
n-docosane,	$C_{22}H_{46}$	230	112.8
n-tetracosane,	$C_{24}H_{50}$	250	125.4
n-pentacosane,	$C_{25}H_{52}$	259	127.8
n-hexacosane,	$C_{26}H_{54}$	268	135.1
n-octacosane,	$C_{28}H_{58}$	286	141.6
n-nonacosane,	$C_{29}H_{60}$	295	145.9
n-hentriacontane,	$C_{31}H_{64}$	312	151.9

Later Francis and Gauntlett[47] examined the oxidation products made by passing a rapid current of air through the original wax at 100°C. for 1200 hours in presence of 5% turpentine. These were found to consist of secondary alcohols, ketones, acids and esters. The secondary alcohols and ketones isolated were found to have the same number of carbon atoms as the first six of the normal hydrocarbons given in the above table. Close examination of crystal spacing of a number of acids obtained/

obtained by the oxidation showed that in all cases these belonged to the normal series of fatty acids.

An examination of waxes from Fushun shale oil and from certain petroleums, was made by Tanaka, Koba-Yashi and Ohno,[48] who found the specimens examined to consist mainly of normal hydrocarbons, and that range for range the wax from shale oil was identical to that from petroleum.

Ferris, Cowles and Henderson[49] cut oil-free wax from petroleum by distillation under vacuum, and by recrystallising from ethylene dichloride succeeded in producing waxes of similar molecular weights, but with widely differing solubilities, melting points and refractive indices. This was taken to be evidence of the presence of isoparaffins. Some of the fractions prepared by Ferris, Cowles and Henderson were subsequently subjected to accurate combustion analyses by Mair and Schicktanz,[50] who came to the conclusion that the samples consisted of normal paraffins and cyclic hydrocarbons.

Clark and Smith[51] by X-ray analysis identified isoparaffins in waxes from Mid Continent crude, and said that at least 20% of the wax consisted of branched chain paraffins.

From the work which has been published on the subject, it would be safe to assume that wax consists mainly/

mainly of normal paraffins with smaller percentages of the branched chain paraffins and cyclic hydrocarbons, although the presence of only the normal hydrocarbons has been proved in the case of waxes from Scotch shale oil.

(2) Importance of Certain Properties on the Utilisation of Waxes.

The chief use of wax is still the manufacture of candles and night lights, but its application for water proofing materials of different kinds is rapidly being extended. It is widely used in the electrical industry as a waterproof coating for cables, etc. and for insulation purposes. Among the more recent uses is that of waxing cartons for food stuffs of all kinds, and also as a coating on certain foods to prevent fermentation and drying. Wax is also largely used in the match industry, in the preparation of floor and furniture polishes and as a fuel for miners' lamps.

Effect of Oil.

Oil is the most common impurity in paraffin wax, and is deleterious in many ways, and for most purposes the oil content should be as low as possible. A notable exception to this is candle making, where the presence of a little oil renders the removal of the candle from the mould easier. Oil, as already observed, is a cause of mottling and opaqueness, and renders waxes not colour stable. The presence of about 1% oil renders a wax mealy or crumbly, and greasy to the touch, and it

can/

can be readily understood that waxes which might come in contact with foodstuffs must be of low oil content.

Oil lowers the tensile strength of wax, and therefore for water proofing and wax films generally the wax should be of low oil content. A desirable property of waxes for this purpose is plasticity.

"Width of Cut".

Plasticity is related to "width of cut", which as described in the section on sweating, depends to a certain extent on the charge to the sweating plant and the degree of sweating carried out. "Width of cut" when applied to oils generally means range of boiling points, but when applied to waxes means range of setting points. Narrow cut waxes are less plastic than longer cut products. Low setting point waxes are more plastic than high setting point waxes, and according to Wilson and Minchin[52], the lower setting point waxes attain a condition of plasticity at lower temperatures relative to their setting point than do waxes of higher setting point. Scotch shale products of setting point below 118°F. show definite plasticity, while those above 125°F. setting point show no plasticity, and to obtain the plasticity and setting point necessary for certain purposes, blends of high and low setting point waxes are prepared.

Narrow cut waxes are necessary for candle making or for moulding purposes generally, because the more/

more plastic products do not leave the moulds readily. Candles made from plastid wax bend in a warm atmosphere. Too high a setting point wax is difficult to light, and therefore narrow cut wax is what is required. With reference to candle manufacture, attention is drawn to an article by Allan[39], which in addition to describing present practice, gives a very interesting history of candles from the time of the early Romans.

Effect of Heat.

In connection with the deleterious effect of oil on waxes generally, it should be remembered that wax is easily oxidised to liquid products on heating, and this is important when paper or cloth is coated with wax by immersion in a bath of hot wax. It is a common practice, in order to obtain a thin uniform film, to heat the wax to too high a temperature, and the wax may be in the bath for very long periods. The effect on the oil content of 135/40 wax of heating for 8 hours at different temperatures is given by Wilson and Minchin[52], and their results are shown in Table 50.

TABLE 50

Temperature of heating	Oil content after heating for 8 hours.
Fresh wax,	0.36%
150°C.	0.39%
200°C.	0.69%
250°C.	1.03%
300°C.	1.67%
350°C.	2.65%

Scotch 122/25 wax, when heated for four

days/

days at a temperature of 100-105°C. increased in oil content from 0.8% to 1.1% and developed a rancid odour and a slight yellow colour.

Scott-Harley in the discussion following a very comprehensive paper by Higgs,[53] on the utilisation of paraffin wax, states that in cases where absence of odour and taste is imperative, the heating area in a wax bath should be as large as possible and the heating medium should be incapable of giving a temperature exceeding 100°C.

The wider application of wax for industrial purposes has necessitated the determination of new physical constants, and in the section which follows, some idea of the tests applied is given.

(3) Analyses of Waxes.

(a) Setting Point.

Setting point determination is the chief test applied to wax, the different grades being prepared to very narrow limits, and until quite recent times setting point and colour determinations were the only tests applied. Throughout the wax industry setting point and melting point mean the same thing, but itis really the setting point which is determined.

There are several methods of determining the setting point of wax, but the only recognised method in this country is that of the Institute of Petroleum.[54] The setting/

setting point is defined as the temperature at which melted wax when allowed to cool under definite specified conditions first shows a minimum rate of temperature change. The standard method of carrying out the test is to place a test tube containing the melted wax in a special water bath with water at 60-80°F. A thermometer is placed in the wax and readings taken every 30 seconds. The setting point is taken as the temperature at which three or more identical consecutive readings are obtained. The accuracy of the test is ±0.2°F.

Calculation of Setting Point for Blending Purposes

Every day in the refinery it is necessary to blend one wax with another to give a blend of definite setting point, and it is generally assumed and found that calculation by simple proportion gives the true result.

Experiments have demonstrated, however, that when blending waxes of widely different setting points this is not strictly correct. Similar results were obtained by Sawyer, Hunter and Nash,[25] and their results are quoted, along with the results for shale waxes, in Table 51.

TABLE 51/

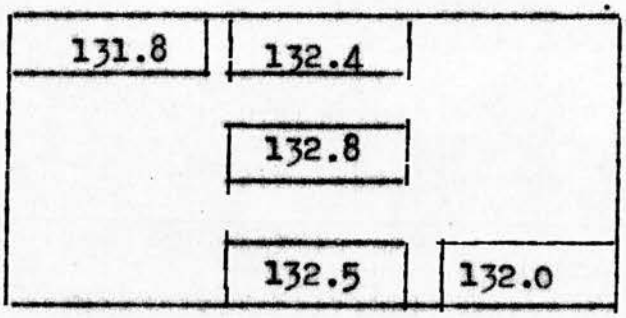
TABLE 51

Shale Waxes				Results of Sawyer, Hunter & Nash using Commercial Waxes.			
% High Sett. Point.	Sett. Point Actual °F.	Sett. Point Calculated. °F.	Difference between Actual & Calcd. °F.	% High Sett. Point	Sett. Point Actual °F.	Sett. Point Calculated. °F.	Difference between Actual & Calcd. °F.
100	132.2	-	-	100	130.95	-	-
				90	129.15	128.7	+ 0.45
75	126.4	125.8	+ 0.6	80	126.95	126.4	+ 0.55
				70	124.9	124.2	+ 0.7
50	120.75	119.45	+ 1.30	50	120.4	119.65	+ 0.75
				40	118.0	117.4	+ 0.6
25	113.75	113.1	+ 0.65	30	115.65	115.1	+ 0.55
				15	112.1	111.75	+ 0.35
Nil	106.7	-	-	Nil	108.35	-	-

Sampling of Wax for Setting Point Determination.

Experiments have shown that solid cakes of either shale wax or petroleum wax when cut horizontally into three equal portions have higher setting points in the centre than at the top or bottom. This takes place with all grades from the highest setting point to the lowest, and generally the centre portion is 1 to 2°F. higher in setting point than either top or bottom. By cooling very slowly a more homogeneous cake is produced.

Wilson and Minchin[52], describing the difficulty of sampling a cake of wax, give the following diagram to illustrate the variations obtainable at different parts of the cake.



The above illustrations show how misleading results could be arrived at by bad sampling, and to obtain a true sample the whole or half of a cake should be melted and a portion of the liquid collected as a sample.

(b) Transition Points.

Carpenter[37], when taking observations on the cooling of wax and its crystallisation from solvents, found evidence to indicate the existence of a transition point between two crystal forms, 18°F. to 27°F. below the setting point. Above the transition point needle shaped prisms were stable, while below the transition point rhomboid plates were obtained. The tests applied by Carpenter to fix the transition point included the determination of solubility in solvents, expansion and density, and air solubility in wax.

The existence of transition points in pure normal hydrocarbons has been demonstrated by means of X-ray and by optical observation by several workers; isoparaffins show no transition effect. Piper[55] gives the relationship

of/

of setting point and transition point with the number of carbon atoms, and shows that as the length of the hydrocarbon chain increases the difference between setting point and transition point decreases.

Garner[56] records the heat of crystallisation and transition for pure hydrocarbons, and Scott-Harley[40] determined transition points in commercial waxes by thermometric methods and found that some waxes show no transition point.

Lord[41] found that the Institute of Petroleum setting point apparatus could be used to determine transition points. The water jacket was filled with water at a temperature of 45 ±0.2°F. and the temperature of the wax during cooling was recorded every half minute from a few degrees above the setting point to approximately 70°F. From the primary cooling curves obtained, inverse rate curves were plotted, and from these transition points were deduced. The plotting of inverse rate curves accentuates the point of inflection in the normal cooling curve and enables the transition temperature to be accurately determined.

This method has been applied to shale waxes and definite evidence of the existence of transition points demonstrated. For commercial wax of setting point 131.5°F. a transition point 37.5°F. below the setting point was obtained/

obtained (Fig.12), while for 135/40 grade, setting point 135.5°F., the transition point was 38°F. below the setting point (Fig.13). For comparison with these a sample of Burmah Oil Coy. 135/40 wax was also tested. This had a setting point of 136.5°F. and gave a transition point 30.5°F. below this temperature. The curves for this wax (Fig.14) very closely resemble those given by Lord for the same grade. Lord found the transition point 31°F. below the setting point.

The transition effect in the shale waxes tested was not so pronounced as in the case of the petroleum wax, and since Scott-Harley and Lord stated that transition effects were more evident in narrow cut waxes, a narrow cut shale wax of setting point 136°F. was prepared by sweating in the laboratory. The curves for this wax are very similar to those for the Burmah wax, the difference between setting point and transition point being 32.5°F. (Fig.15)

The results confirm Lord's conclusions that narrow cut wax gives a more definite transition point than a wider cut product, and that the difference between setting point and transition point decreases with narrowness of cut. In narrow cut wax prepared by vacuum distillation, Lord showed a difference of only 21°F. between setting point and transition point.

Scott-Harley/

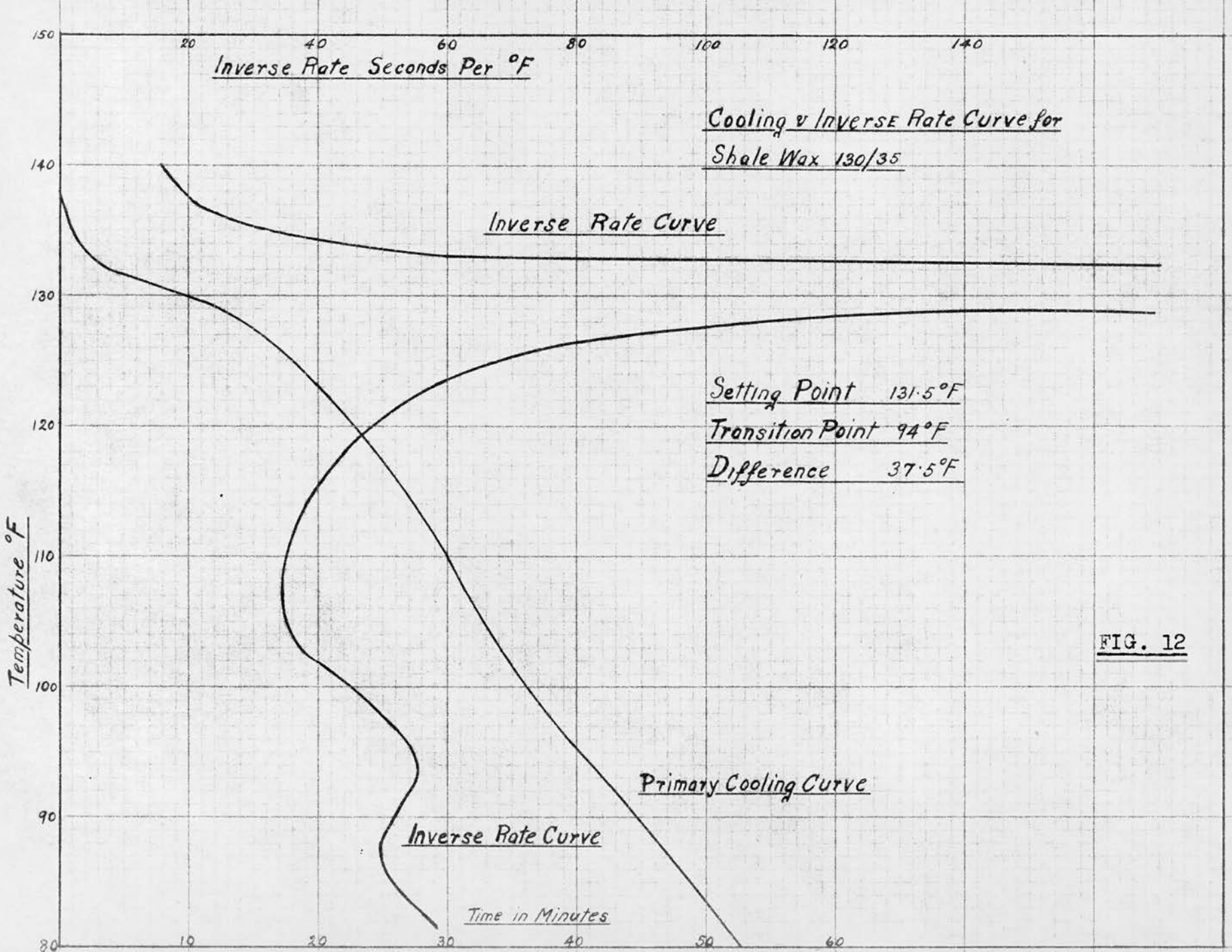

Inverse Rate Seconds Per °F
20
40
60
80
100
120
140
Cooling v Inverse Rate Curve for
Shale Wax 130/35
Inverse Rate Curve
Setting Point 131·5°F
Transition Point 94°F
Difference 37·5°F
Primary Cooling Curve
Inverse Rate Curve
Time in Minutes
Temperature °F
150
140
130
120
110
100
90
80
10
20
30
40
50
60

FIG. 12

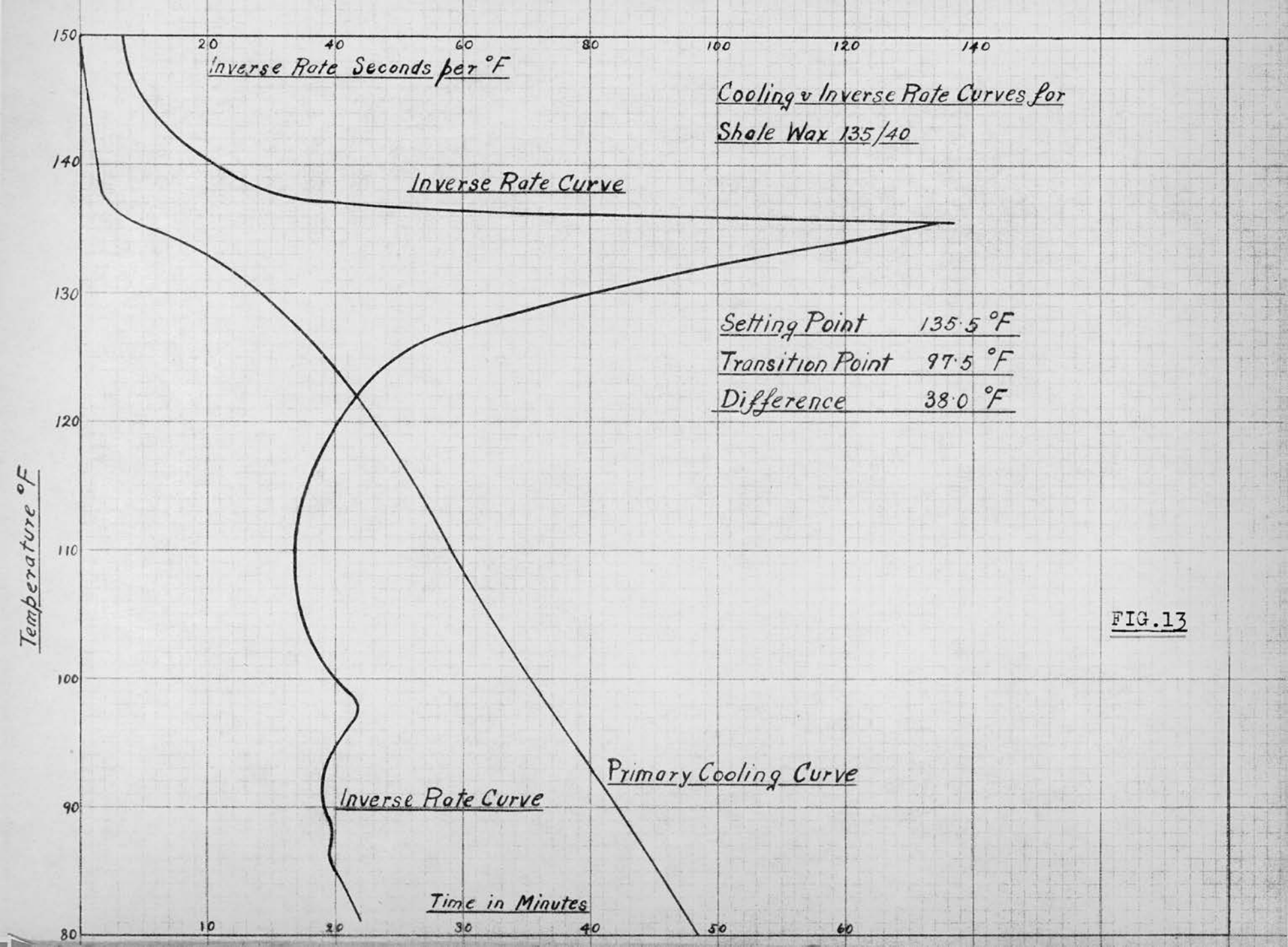

Inverse Rate Seconds per °F
20
40
60
80
100
120
140
Cooling v Inverse Rate Curves for
Shale Wax 135/40
Inverse Rate Curve
Setting Point 135·5 °F
Transition Point 97·5 °F
Difference 38·0 °F
Temperature °F
150
140
130
120
110
100
90
80
Primary Cooling Curve
Inverse Rate Curve
Time in Minutes
10
20
30
40
50
60

FIG.13

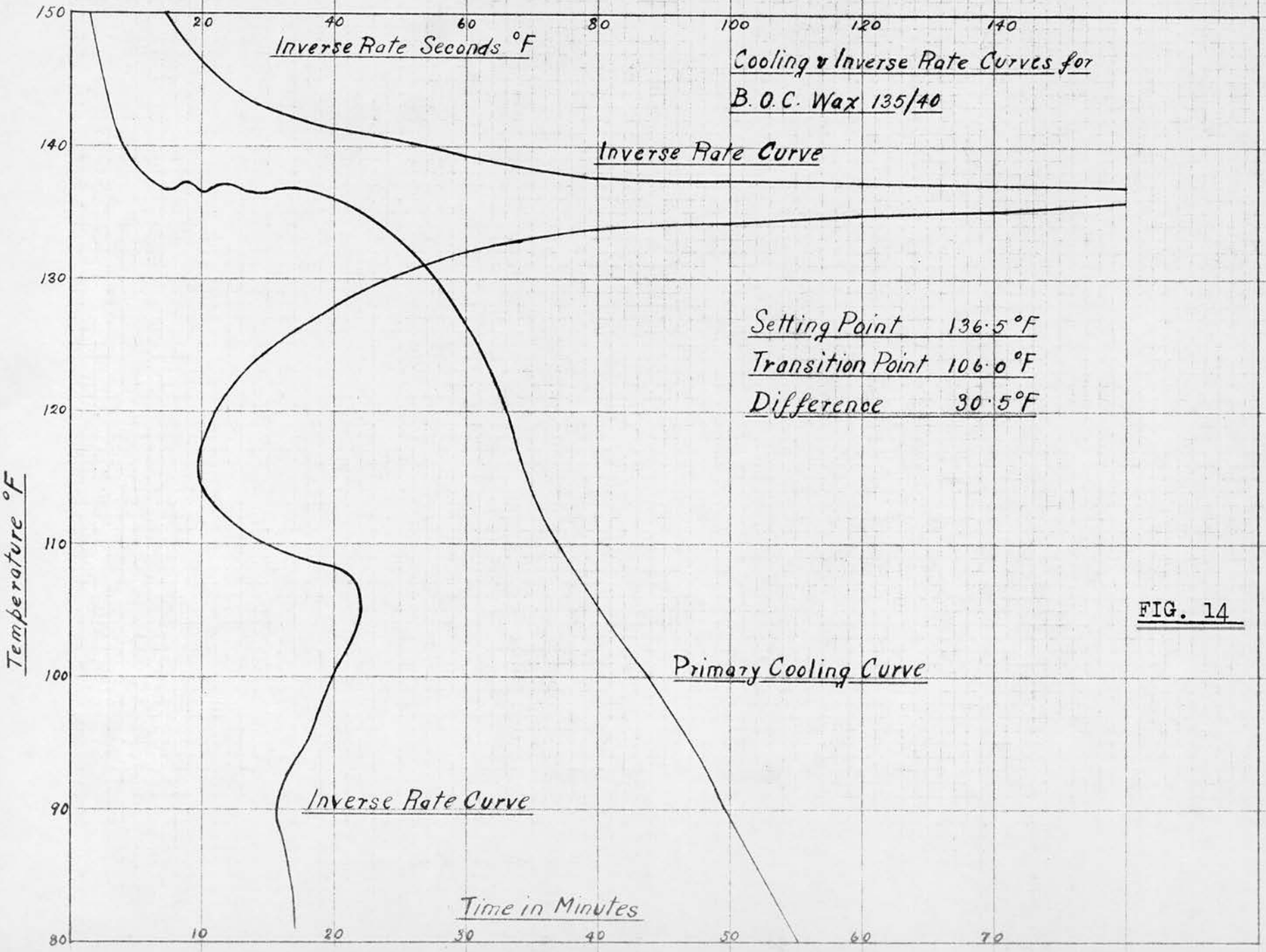

Inverse Rate Seconds °F
Cooling & Inverse Rate Curves for
B. O. C. Wax 135/40
Inverse Rate Curve
Setting Point 136·5 °F
Transition Point 106·0 °F
Difference 30·5 °F
Primary Cooling Curve
Inverse Rate Curve
Temperature °F
Time in Minutes
150
140
130
120
110
100
90
80
20
40
60
80
100
120
140
10
20
30
40
50
60
70

FIG. 14

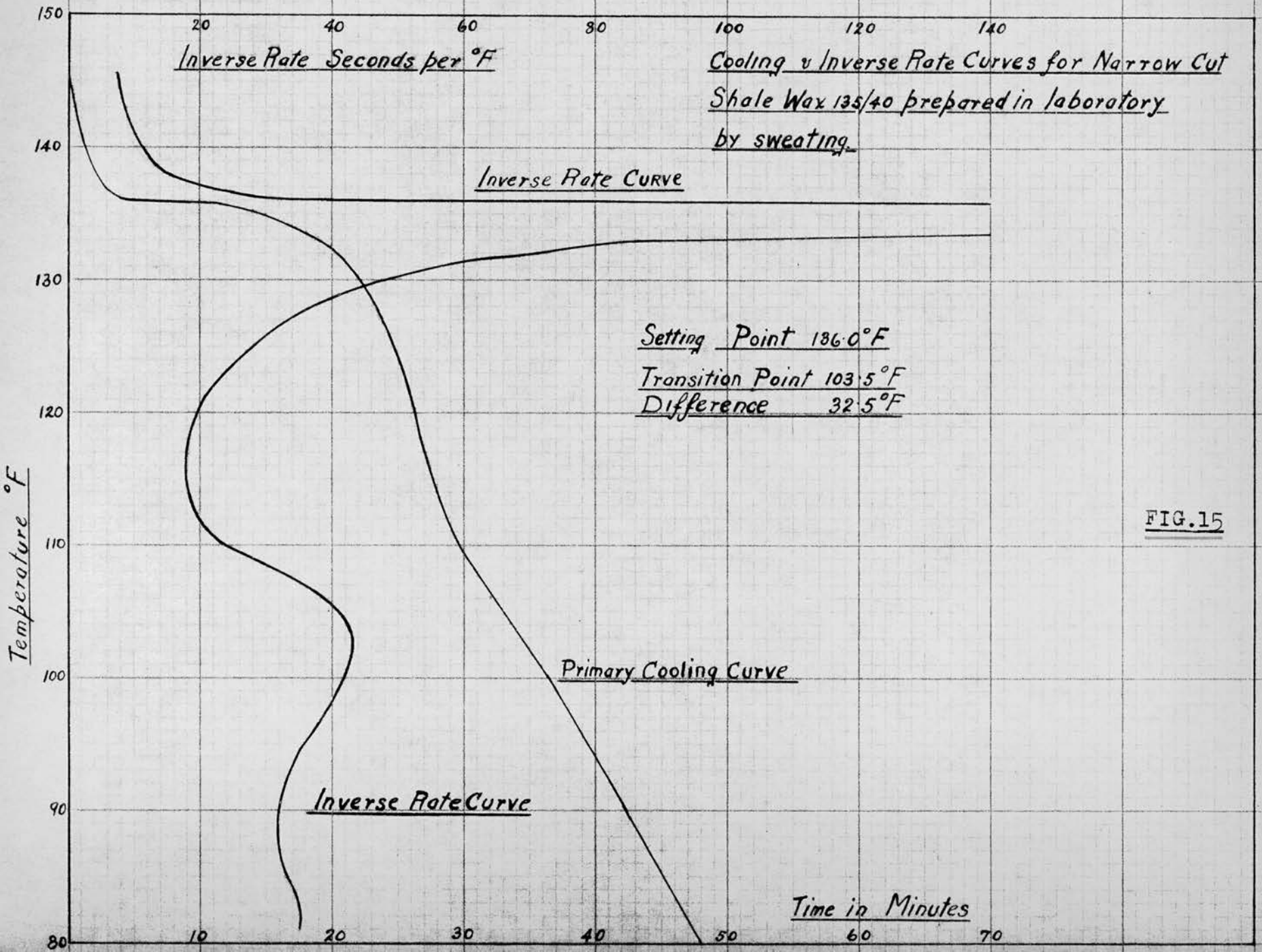

Inverse Rate Seconds per °F
Cooling v Inverse Rate Curves for Narrow Cut Shale Wax 135/40 prepared in laboratory by sweating.
Inverse Rate Curve
Setting Point 136·0°F
Transition Point 103·5°F
Difference 32·5°F
Primary Cooling Curve
Inverse Rate Curve
Temperature °F
Time in Minutes
150
140
130
120
110
100
90
80
20
40
60
80
100
120
140
10
20
30
40
50
60
70

FIG.15

Scott-Harley, in duscussing the practical application of transition effect, considered that waxes which do not show transition effect should be more suitable for waxing paper and for certain electrical purposes, because they are more homogeneous in structure and more stable in a changing temperature. On the other hand the contraction which takes place may be beneficial when wax is moulded as in candle making. A wax showing transition effect would leave the mould more readily than a wax which showed no transition point. The presence or otherwise of transition points could therefore be used as an indication of the use to which certain grades of wax should be put.

(c) Penetration and Hardness.

Description of Jackson-Burmah Wax Testing Machine and Method of Operation.

The Jackson-Burmah wax testing machine[57] was designed by the Research Staff of the Burmah Oil Co. to test the hardness of paraffin waxes.

The machine consists essentially of a beam, which hangs freely on a pivot at one end. A known weight is attached to the other end, and at the centre an indenting piece, which is normally a 60° cone, is fixed. A test cake of wax is attached to an adjustable block, and the position of the block relative to the indenting piece can be varied by means of a screw. This is used to bring the/

the sample and cone into contact. When contact has been made a reading is taken on a scale, and the position of the loaded beam is then altered in such a way that the indenting cone is pushed into the wax under specified conditions as regards time and applied load. The depth of penetration is measured in cms. The test sample is moved slightly to bring another position on the cake opposite the cone, and further penetrations carried out. As a rule six such determinations are sufficient, and the average result is taken. The whole apparatus is enclosed in a water bath so that determinations can be made at definite temperatures.

The sample to be tested is melted and a known weight put into a nickel basin and allowed to cool to atmospheric temperature. The sample is cooled for at least 16 hours before putting into the machine, and is then allowed to stand in the machine for one hour at the temperature of the test before any readings are taken.

Interpretation of Results.

The method of testing and the interpretation of results were the work of the staff of the Burmah Oil Co., who found that for commercial grade waxes of setting point above 122°F. the hardness at 60°F. was independent of the setting point, and was influenced by the addition of relatively small quantities of oil. All oil-free waxes of setting point above 122°F. were found to have the same hardness, and the addition of a given quantity of/

of oil lowered the hardness to the same degree in each case. Later it was found that for waxes of setting point below 122°F. the hardness at 37.5°F. was a function of the oil content. These facts were then used to develop a means of determining oil in wax, for which there had never previously been a satisfactory method. Graphs showing the relationship between penetration in cms. and oil content of wax were drawn by testing waxes of known oil contents. These were prepared by adding oil to oil-free wax, oil-free wax being wax which on sweating showed no increase in hardness.

The hardness at higher temperatures, as well as being influenced by oil content, depended on the proportion of low setting point wax present in the sample. It was found that hardness approached zero not at the setting point but at the transition point, which was normally 25/35°F. below the setting point. Consequently by determining the hardness at a temperature 40°F. below the setting point, a comparative measure of "width of cut" could be obtained.

The results of tests at the lower temperatures are recorded as oil content, % by wt., while at the higher temperatures the penetration is calculated to Hardness No. as under:-

$$\text{Hardness Number} = \frac{\text{Applied load in kg.}}{\text{surface area of impression}}$$

Since/

Since the indenting piece is a 60° cone, and applied load is normally 1.5 kg., Hardness Number = $\frac{1.5}{2/3\pi h^2}$ where h represents depth of penetration in cms.

Uses of Machine.

The chief use of the machine is as a routine control over refining operations. As a rule, the oil content of a wax should be as low as possible, and the hardness number as high as possible. These are both achieved by suitable sweating, and the machine is the only method of checking the product. In some cases products of higher oil contents than usual are desired, and in other cases where plastic waxes are demanded, the hardness number must be low, and the machine is useful in the preparation of such grades.

In investigating complaints regarding consignments, or matching competitive products, the more physical constants that can be determined on the wax the better, and hardness tests are an advantage in this respect.

The machine has also been found useful for testing products from experimental work, particularly on sweating, where oil contents are required. In the case of fractions having oil varying from about 3% to 10%, these are blended with oil-free wax in such a proportion that the blend contains 1 to 2% oil, and so gives a reasonable reading on the graph.

Determination/

Determination of Oil Content.

Oil-free wax of setting point 131°F. was prepared by repeated sweating of shale wax of setting point 118°F. until further sweating gave no change in penetration at 60°F. Known quantities of oil, of same grade as would normally be present in wax, were added to this oil-free wax and penetration at 60°F. determined after each addition. The results are shown graphically in Fig.16.

The effect on penetration at 37.5°F. of adding oil to oil-free wax of setting point 115.3°F. prepared by prolonged sweating of wax of setting point 106°F. is also given in Fig.16. These two curves are used to convert the penetration at 60°F. for high setting point grades and at 37.5°F. for lower setting point waxes into oil content (% by weight).

Blends of 106/8 and 130/35 waxes were prepared and the oil contents determined by means of the graphs. For comparison, the oil contents of the blends were calculated by proportion, taking the oil content of the components from the graphs. Table 52 gives the comparison.

The results show that for blending purposes, the oil content of a blend can be calculated from those of the constituents.

TABLE 52/

TABLE 52

Wax.	Oil content from Graphs, % by weight	Oil content calcd. from oil content of components taken from graphs % by weight.
106/08	1.2	-
106/08 - 75%) 130/35 - 25%)	0.9	0.95
106/08 - 50%) 130/35 - 50%)	0.7	0.7
106/08 - 25%) 130/35 - 75%)	0.4	0.45
130/35	0.2	-

Small quantities of coconut oil are sometimes added to wax when making candles, with the object of making removal from the moulds easier. As a matter of interest, and to find if the hardness test could be used to determine how much oil had been added, known quantities of coconut oil were added to 125/30 wax, and the penetration at 60°F. determined after each addition. The results, shown in Table 53, have been converted to oil content using the graph for the high setting point waxes.

TABLE 53

	Oil content % by weight.
Original 125/30 wax,	0.6
After add'n.of 0.1% coconut oil,	0.8
" " " 0.25% " "	0.9
" " " 0.5% " "	1.1

From/

From these results it can be seen that coconut oil lowers the hardness to the same extent as the mineral oil in the wax, and that by determining the hardness and by using the graph an estimate of the coconut oil added to wax of known oil content could be made.

Effect of Temperature on Hardness
(Determination of "Width of Cut" and Plasticity)

The penetration of various grades of shale waxes has been determined at different temperatures from 40°F. till the hardness approached zero. The results are given in Fig.17, which shows penetration in cms. against temperature, and hardness number (calculated from the penetration in cms.) against temperature. An interesting point in these curves is that in all cases the hardness approached zero at a temperature approximately 30°F. below the setting point of the wax.

The hardness number/temperature curves for waxes of setting point 115/18°F. and below, show a different slope than those for the higher setting point waxes. These lower setting point waxes are more plastic than the others and have lower hardness numbers at comparative temperatures below the setting point. See Table 54.

TABLE 54

	106/8	110/15	115/18	118/20	122/25	125/30	130/35
Setting Point, °F.	106.7	111.2	117.2	119.3	124.2	129.1	132.2
Hard.No.40°F.below Set.Pt.	3.6	4.2	2.2	3.8	5.0	5.2	6.4
" " 50°F. " "	12	14	12	14	20	18	30
" " 60°F. " "	28	34	26	38	40	36	54

Various/

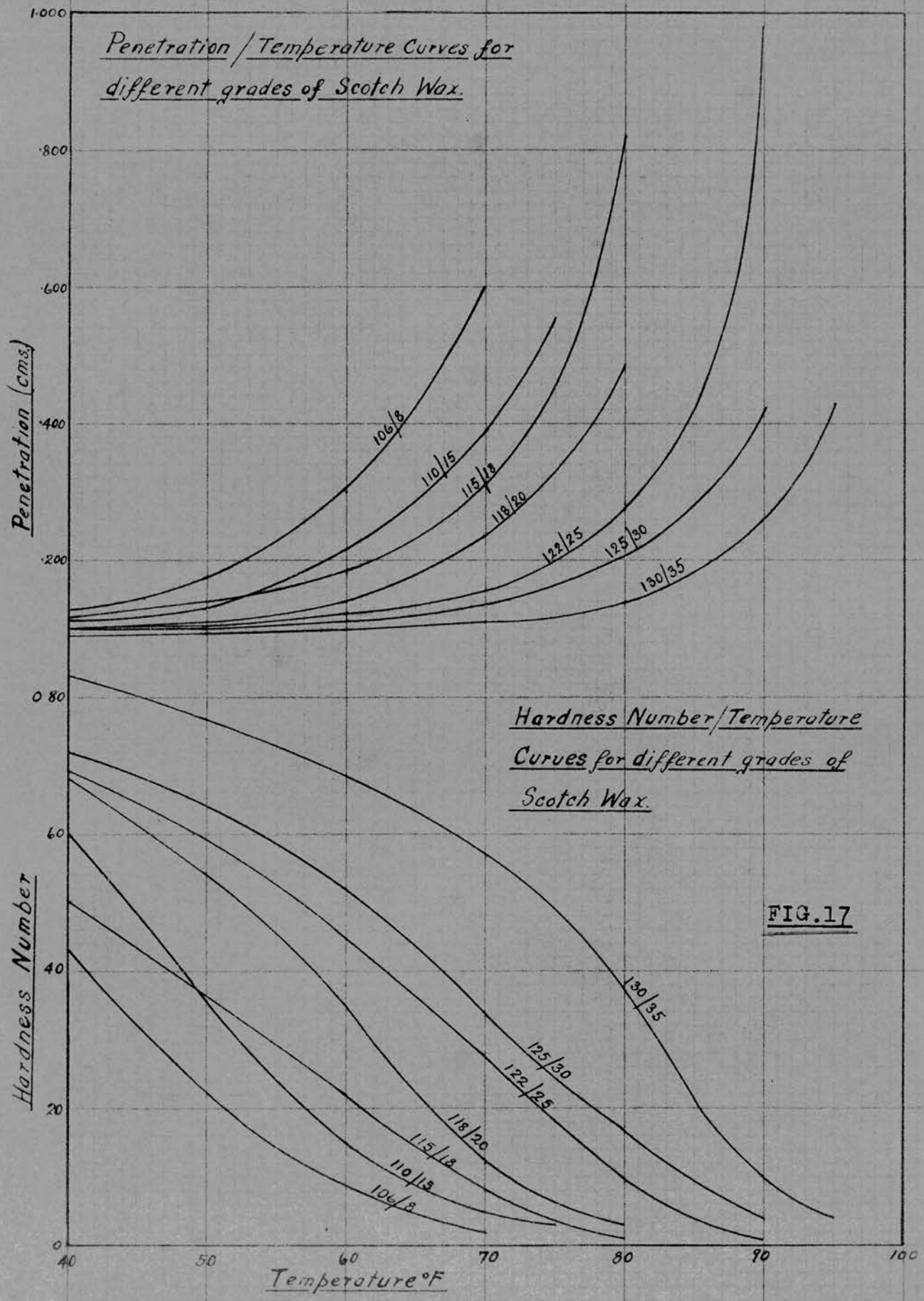

Penetration / Temperature Curves for different grades of Scotch Wax.
Penetration (cms.)
1.000
.800
.600
.400
.200
106/8
110/15
115/18
118/20
122/25
125/30
130/35
Hardness Number / Temperature Curves for different grades of Scotch Wax.
Hardness Number
0 80
60
40
20
0
130/35
125/30
122/25
118/20
115/18
110/15
106/8
40
50
60
70
80
90
100
Temperature °F

FIG. 17

Various amounts of oil from 0.25% to 4.0%, have been added to oil-free wax, and hardness number/ temperature curves drawn for the mixtures. These graphs, Fig.18, show that as oil is increased the curve becomes flatter. Since plastic waxes have a steep hardness number/ temperature cruve, the addition of oil has reduced plasticity. This is in agreement with actual experience that oil destroys plasticity and makes waxes crumbly and mealy.

Blends of 106/8 wax and 130/35 wax were prepared, and hardness number/temperature curves made for each blend, and these are given in Fig.19. The addition of the low setting point wax lowered the hardness to an extent depending on the percentage of low setting point constituent in the blend. The blend consisting of 75% 130/35 and 25% 106/8 has given an entirely different curve from the original 130/35 wax, the former being much steeper, indicating a wider cut wax and showing evidence of plasticity.

A comparison of the hardness at temperatures 40, 50 and 60°F. below the setting point for the blends marked 2, 3 and 4, on the hardness number/temperature curves (Fig.19) with the hardness at corresponding temperatures for the commercial grades of like setting point (Fig.17) is given in Table 55.

TABLE 55/

Effect of oil on Hardness Number / Temperature Curve

1. 4.0% oil added to oil free Wax.
2. 3.0% " " " " " "
3. 2.0% " " " " " "
4. 1.5% " " " " " "
5. 1.0% " " " " " "
6. 0.75% " " " " " "
7. 0.50% " " " " " "
8. 0.25% " " " " " "
9. Oil free Wax.

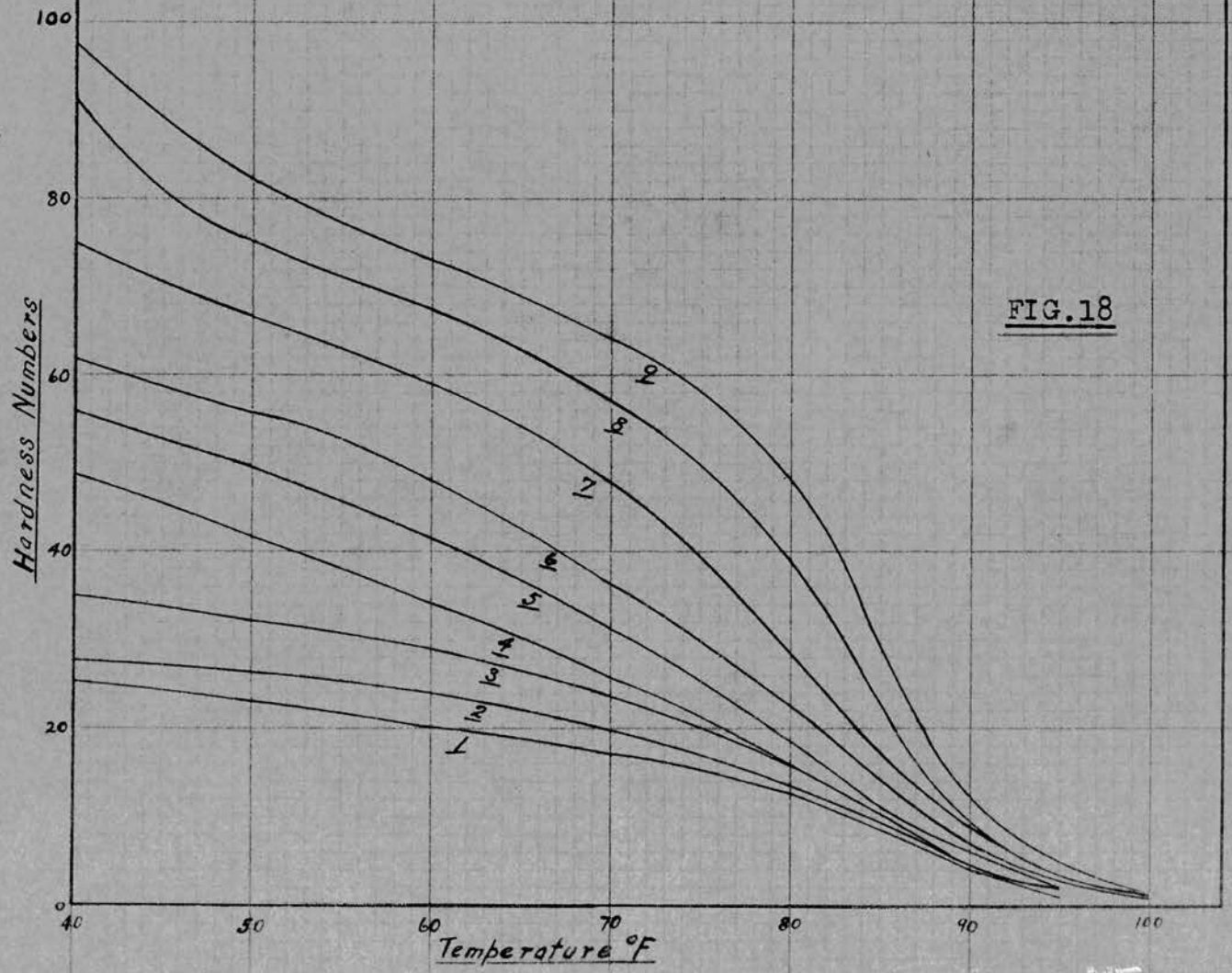


FIG. 18

TABLE 55

	106/8 75% 130/35 25%	Comm'l. 110/15	106/8 50% 130/35 50%	Comm'l. 118/20	106/8 25% 130/35 75%	Comm'l. 125/30
Setting Pt., °F.	113.75	111.2	120.75	119.3	126.4	129.1
Oil content, %	0.9	0.9	0.7	0.7	0.4	0.7
Hard.No. 40°F.below St.Pt.	1.5	4.5	0.5	3.5	2	5
" " 50°F " "	8	13.5	7.5	14.0	9	18
" " 60°F." "	23.5	34	18	38	23.5	36

These results show in every case that the blended wax has a decidedly lower hardness than the corresponding commercial grade at a comparative temperature. From the oil content of the waxes it will be seen that this effect is not due to higher oil contents, and therefore these tests prove that hardness number/temperature curves do give a measure of "width of cut".

Calculation of Hardness Numbers of Blends.

Frequently in the refinery blends of high and low setting point waxes are prepared, and it has already been shown that setting points and oil contents for the blend can be calculated. The only other constant which it would be an advantage to be able to calculate would be hardness number, in order that wax of desired "width of cut" could be made. The Burmah Oil Coy. found that by applying the logarithmic mixture law a reasonable forecast of hardness number at a definite temperature could be made. The formula is:-

x/

$x \log H_1 + (100 - x) \log H_2 = 100 \log H_m$ where x = % by wt. of wax of hardness H_1 at the desired temperature. H_2 is the hardness of the other constituent and H_m is hardness of the blend.

The hardness numbers of blends of Scotch 106/8 wax and Scotch 130/35 wax were determined at different temperatures, and a comparison between the actual results and the hardness numbers calculated from the above formula is shown in Table 56.

TABLE 56

Temp. °F.	106/8 Wax	130/35 Wax	106/8, 25% 130/35,75%		106/8, 50% 130/35,50%		106/8, 75% 130/35,25%	
			Calculated	Actual	Calculated	Actual	Calculated	Actual
40	43	84	71	83	60	70	51	60
50	23	76	56	61	42	46	31	35
60	8.2	68	40	36	24	19	14	12.5
70	2.0	57	24	16	10.7	8.6	4.6	4.3

Fig.20 shows on a logarithmic scale the effect of blending on hardness. At any temperature the curve showing variation in hardness with composition is roughly a straight line, and so the linear logarithmic relationship given above is accurate enough to calculate approximate hardness numbers.

Effect of Stearine on Hardness.

Candles are frequently made from a mixture of Stearine and wax. The reasons for adding Stearine are: (1) to give an opaque candle, which is generally desired/

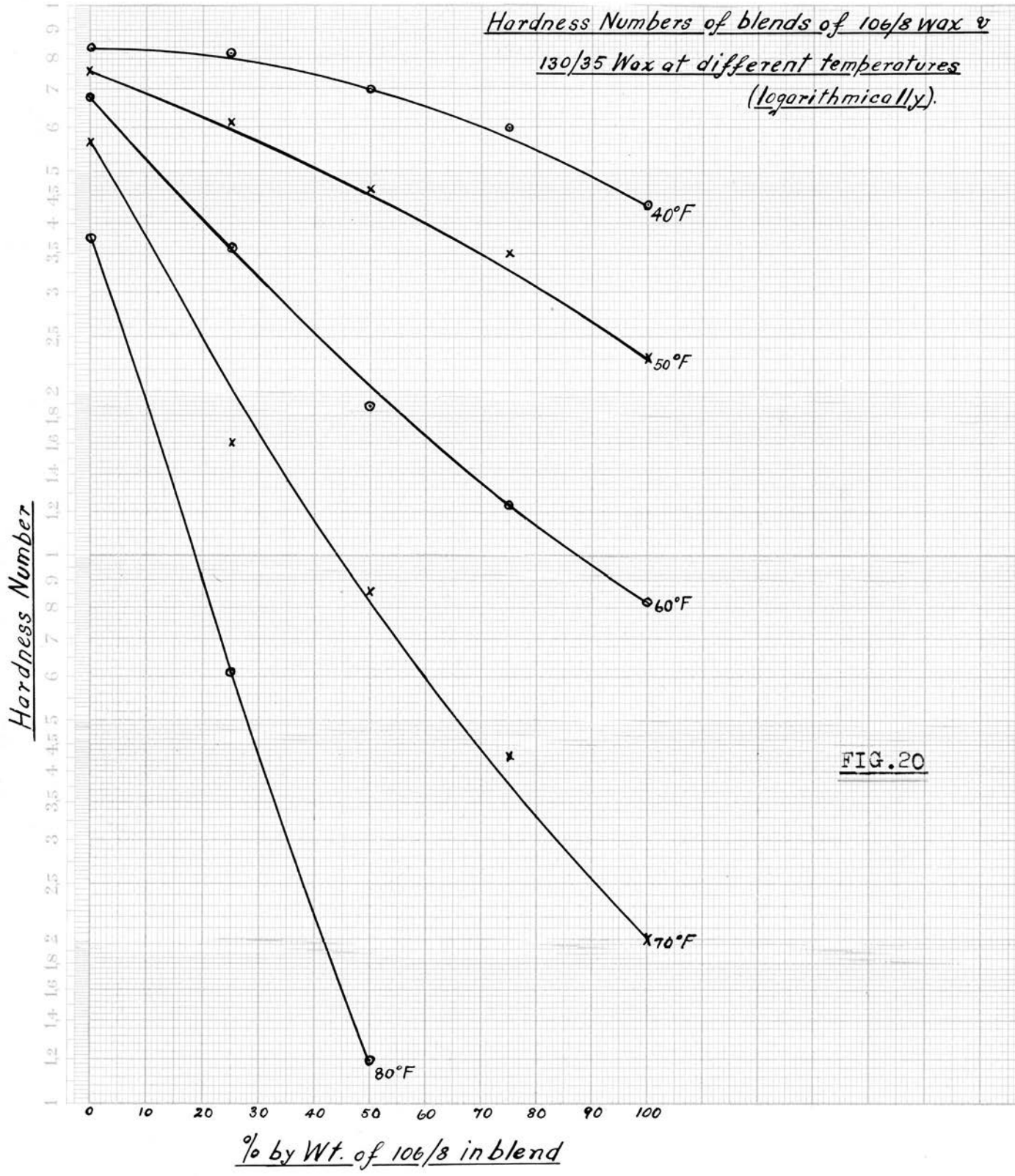

Hardness Numbers of blends of 106/8 wax v
130/35 Wax at different temperatures
(logarithmically).
40°F
50°F
60°F
70°F
80°F
Hardness Number
% by Wt. of 106/8 in blend
0
10
20
30
40
50
60
70
80
90
100

FIG. 20

desired, (2) to prevent mottling, and (3) to give a candle which can be used at higher temperatures without bending than would be possible if no Stearine was added.

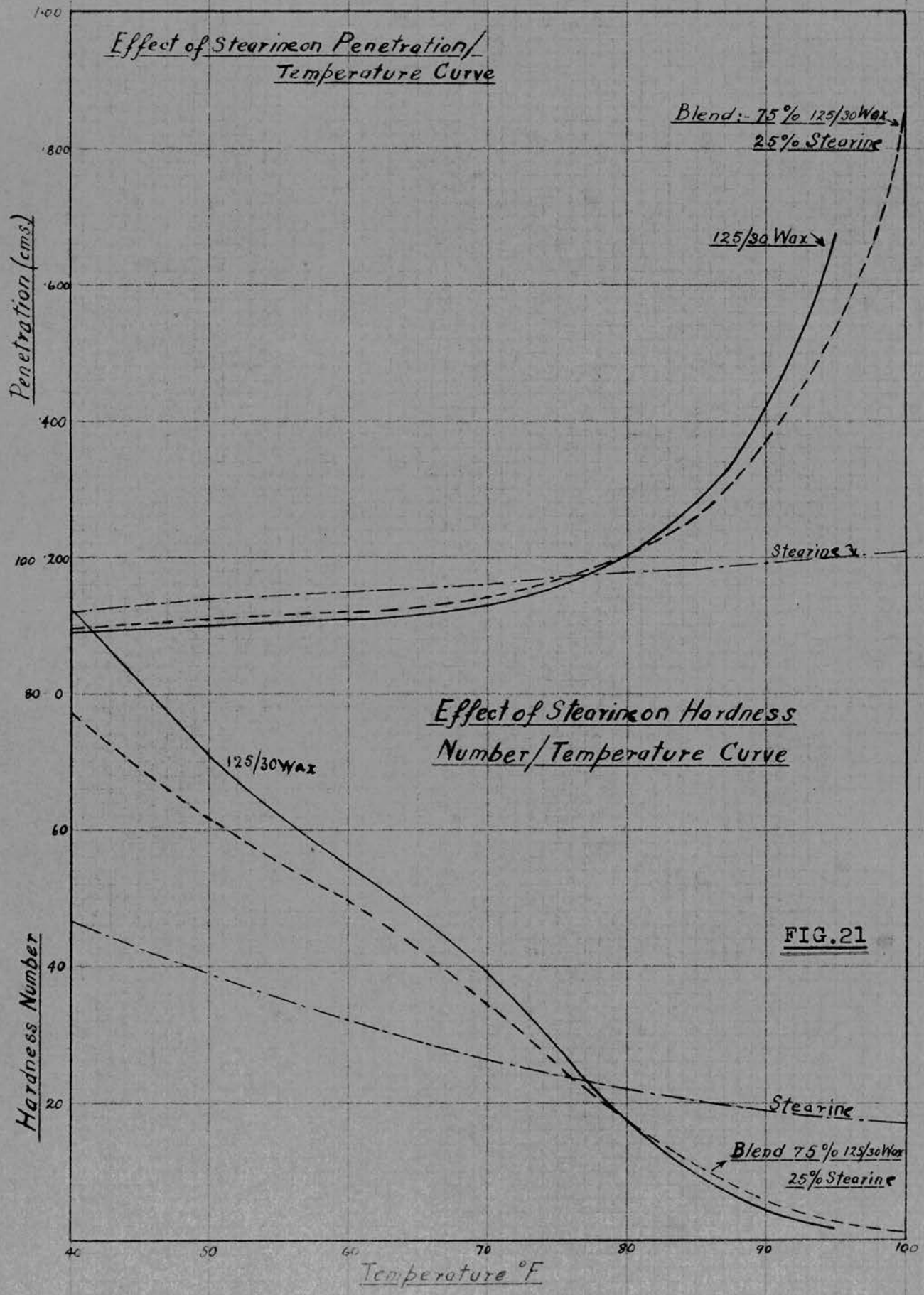
A common grade of candle is made from a blend of 125/30 shale wax 75%, and Stearine 25%. Hardness number/temperature curves for the wax, Stearine and the 75/25 blend have been drawn (Fig.21). The Stearine shows a very flat curve, and its effect on wax is to lower the hardness at low temperatures, and increase the hardness at higher temperatures. This increased hardness at the higher temperatures would reduce the tendency for the candle to bend, which as already stated is one of the reasons for adding Stearine.

The hardness numbers at different temperatures for the 75/25 wax/Stearine blend were calculated by the logarithmic mixturelaw, and the results, as shown in Table 57, are very near to those actually determined.

TABLE 57

Temp. °F.	HARDNESS NUMBERS			
	125/30 Wax.	Stearine	125/30 Wax, 75% Stearine, 25%	
			Calculated	Actual.
40	92	47	78	78
50	71	35	59	60
60	54	34	48	50
70	42	24	36	35
80	16.5	25	18.3	16.5
85	8.8	18	11.0	10.8
90	4.0	21	6.0	5.1

Conclusions/


Effect of Stearine on Penetration/ Temperature Curve
Blend:- 75% 125/30 Wax 25% Stearine
125/30 Wax
Stearine
Penetration (cms.)
1·00
·800
·600
·400
·200
0
Effect of Stearine on Hardness Number/Temperature Curve
125/30 Wax
Stearine
Blend 75% 125/30 Wax 25% Stearine
Hardness Number
80
60
40
20
40
50
60
70
80
90
100
Temperature °F
FIG.21

Conclusions from Experiments with Shale Waxes.

The experiments made show that the hardness test as determined by the Jackson-Burmah machine gives a reliable measure of oil content and "width of cut".

Oil content in waxes of setting point above 122°F. is determined by finding penetration at 60°F. while for waxes of setting point below 122°F. penetration is determined at 37.5°F. In both cases the penetration can be converted to oil content (% by weight) by reference to Fig.16.

Hardness/temperature curves for all grades of wax show that hardness approaches zero at a temperature approximately 30°F. below the setting point of the wax.

The hardness at a temperature 40°F. below the setting point can be used as a test for "width of cut". A wax containing constituents of widely varying setting points gives lower hardness at this temperature than a narrower cut wax.

Plastic waxes have steeper hardness/temperature curves than non-plastic waxes. The presence of oil gives a flat hardness/temperature curve.

The hardness number of a blend of two waxes can be approximately calculated from those of the constituents by applying the logarithmic mixture law.

It is shown that the addition of 25% Stearine/

Stearine to 125/30 wax lowers the hardness at lower temperatures and increases it at higher temperatures. Stearine has a very flat hardness/temperature curve. Calculation of hardness numbers at different temperatures for 75/25 Wax/Stearine blend by the logarithmic mixture law gave results very similar to those actually determined.

(d) Analyses of Various Grades of Waxes from Shale Oil.

TABLE 58

Grade	130/35	125/30	122/25	118/20	110/15	106/8
Sett.Pt.(I.P.T.)°F.	132.2	129.1	124.2	119.3	111.2	106.7
Sp.Gr. at 150°F.	.776	.776	.775	.774	.773	.772
Distillation Test.						
I.B.P.	345°C.	345°C.	345°C.	338°C.	335°C.	330°C.
10% dist. at,	372 "	372 "	368 "	360 "	352 "	345 "
20% " "	-	-	373 "	367 "	361 "	352 "
30% " "	-	-	-	371 "	366 "	357 "
40% " "	-	-	-	-	370 "	363 "
50% " "	-	-	-	-	375 "	368 "
60% " "	-	-	-	-	-	375 "
% dist. at 350°C.	0.5	0.5	0.5	1.5	5.0	15.0
" " " 375 "	16.5	18.5	25.0	38.0	50.0	60.0
Flash Point, °F. (P.M. closed)	395	400	390	380	370	360
Aniline Point, °C.	116.5	115.2	113.7	112.2	109.7	108.2
Oil Content, %	0.2	0.7	1.0	0.7	0.9	1.2
Hardness No. at 40°F. below setting point,	6.4 @ 92°F.	5.2 @ 89°F.	5.0 @ 84°F.	3.8 @ 79°F.	4.2 @ 71°F.	3.6 @ 67°F.

Comments on Analyses of different Grades of Waxes.

From Table 58 it will be seen that the specific gravity decreases as the setting point decreases. The 132.2°F. wax had a specific gravity at 150°F. of .776, and/

and the 106.7°F. wax had a specific gravity of .772.

Lord[58] gives the following formula for calculating the specific gravity at 65°C. (149°F.) of waxes from the setting point:-

Specific Gravity = 0.775 + (S.P.°F. - 120) 0.0003

The specific gravities of various shale waxes calculated by this formula are compared in Table 59 with the actual values determined experimentally.

TABLE 59

Sett.Point, °F.	132.2	129.1	124.2	119.3	111.2	106.7
Actual Sp.Gr.	.776	.776	.775	.774	.773	.772
Calcd. Sp.Gr.	.7787	.7777	.7763	.7748	.7724	.7710

The calculated specific gravity for higher setting point wax is slightly higher than the actual specific gravity, while for the low setting point wax the calculated figure is slightly lower than the actual.

Although there is considerable overlapping in the distillation ranges of the different grades, the lower the setting point the more volatile the wax. The wax, setting point 106.7°F., showed 15% distilling at 350°C. and 60% distilling at 375°C. as compared with the 132.2°F. wax, which had 0.5% distilling at 350°C. and 16.5% at 375°C.

The flash point decreases as the setting point of the wax decreases. The 130/35 wax had a flash point of 395°F. and the 106/8 wax a flash point of 360°F.

The/

The aniline point shows a gradual decrease from 116.5°C. for 130/35 wax to 108.2°C. for 106/8 wax. The relationship between aniline point and setting point has been deduced by Lord[58] and found to be:-

Aniline Point °C. = 0.319 x Sett.Pt.(°F) + 75.6

The aniline points for shale waxes when calculated from the setting point by the above formula compared with the value determined by experiment are given in Table 60.

TABLE 60

Sett.Point, °F.	132.2	129.1	124.2	119.3	111.2	106.7
Actual Aniline Point, °C.	116.5	115.2	113.7	112.2	109.7	108.2
Calcd. Aniline Point, °C.	117.8	116.8	115.2	113.6	111.1	109.6

These aniline points are slightly higher in every case than the determined results, and if the figure 75.6 was replaced by 74.2, the actual and calculated aniline points would agree to within 0.2°C. in every case.

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REFINING OF SHALE OIL IN SCOTLAND

By

G. H. SMITH

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REFINING OF SHALE OIL IN SCOTLAND.*

By G. H. Smith,* Ph.D., M.Inst.Pet., and W. B. Peutherer,* F.I.C., M.Inst.Pet.

Summary.

Refining of shale oil in Scotland has been directed at different periods to the production of various primary products. In the early days of the industry burning oil was the main objective of the refiner, but as the industry developed, the refining of shale oil aimed at the production of a full range of products—motor spirit, solvent naphtha, burning oil, gas oil, wax, light lubricating oil and fuel oil. Such processes have been adequately described in previous publications, and this paper deals with what are to-day the most important products—diesel oil for high-speed compression-ignition engines, motor spirit and wax.

The chemistry of shale oil—particularly Scottish shale oil—is briefly reviewed, but unfortunately only the lightest fractions have been studied to any great extent.

The various refining steps to produce the maximum quantity of diesel oil are indicated, together with the experimental work on which the present refining scheme is based.

Wax extraction and refining, which at the moment is a necessary feature of any such refining scheme, is dealt with in some detail.

Alternative methods for the manufacture of motor spirit from shale oil, with special reference to their effect on octane rating, are discussed.

I. Chemistry of Shale Oil.

Shale oil is a decomposition product of the organic matter in oil shale, but up to the present it has been found impossible to isolate this material. The obvious method of extraction by solvents has proved only partly successful. Gavin and Adyelotte [1] reported the extraction from certain shales by organic solvents of 10·98 per cent. or 55·8 per cent. of the yield of oil by distillation. Hentze,[2] on the other hand, attacked kukersite with the inorganic acids, hydrochloric and hydrofluoric, and then extracted the residue with chloroform, whereby he isolated 72 per cent. of the bitumen in the shale. Bailey [3] obtained a yield of 0·98 per cent. of organic matter (10 per cent. of the distillation yield of oil) by extracting Scotch shale with carbon disulphide and chloroform for 12 days.

By distillation under reduced pressure, McKee and Goodwin [4] obtained from certain shales an intermediate product between the bitumen as it exists in shale and shale oil. This material was highly unsaturated, being completely soluble in sulphuric acid (sp. gr. 1·84). On distillation, decomposition took place, giving oil, 48 per cent. insoluble in acid, and coke. Distillation apparently breaks down the heavy polymers into more saturated hydrocarbons and coke.

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* Scottish Oils, Ltd.

The nature of shale oil, therefore, depends first on the fundamental nature of the organic material, and secondly on the method and rate of retorting.

Shale oils, like petroleum, can be generally classified as paraffin, mixed or asphaltic base, although all contain a considerable proportion of unsaturated hydrocarbons. Scotch, Australian and Fushun (Manchuria) shale oils are examples of the paraffin crudes, whilst Estonian and Tasmanian shale oils fall into the asphaltic class. The spirit fractions of the latter are characterized by the comparatively high aromatic content (13–15 per cent.), whilst the former contain only traces of aromatic compounds.

Hydrocarbons.

As regards the hydrocarbon content of Scotch shale oil, little information is available outside the motor-spirit range. An analysis of shale spirit gives the following composition :—

Paraffins	42·5%
Naphthenes	9·5%
Unsaturated hydrocarbons	48·0%

Steuart [5] isolated pentane and *iso*pentane, hexanes and heptanes, and identified the naphthenes, methyltetramethylene, pentamethylene, hexamethylene and methylhexamethylene.

Sulphur.

The sulphur content of Scotch shale oil is low (0·45 per cent.) and its distribution is shown in Table I.

TABLE I.

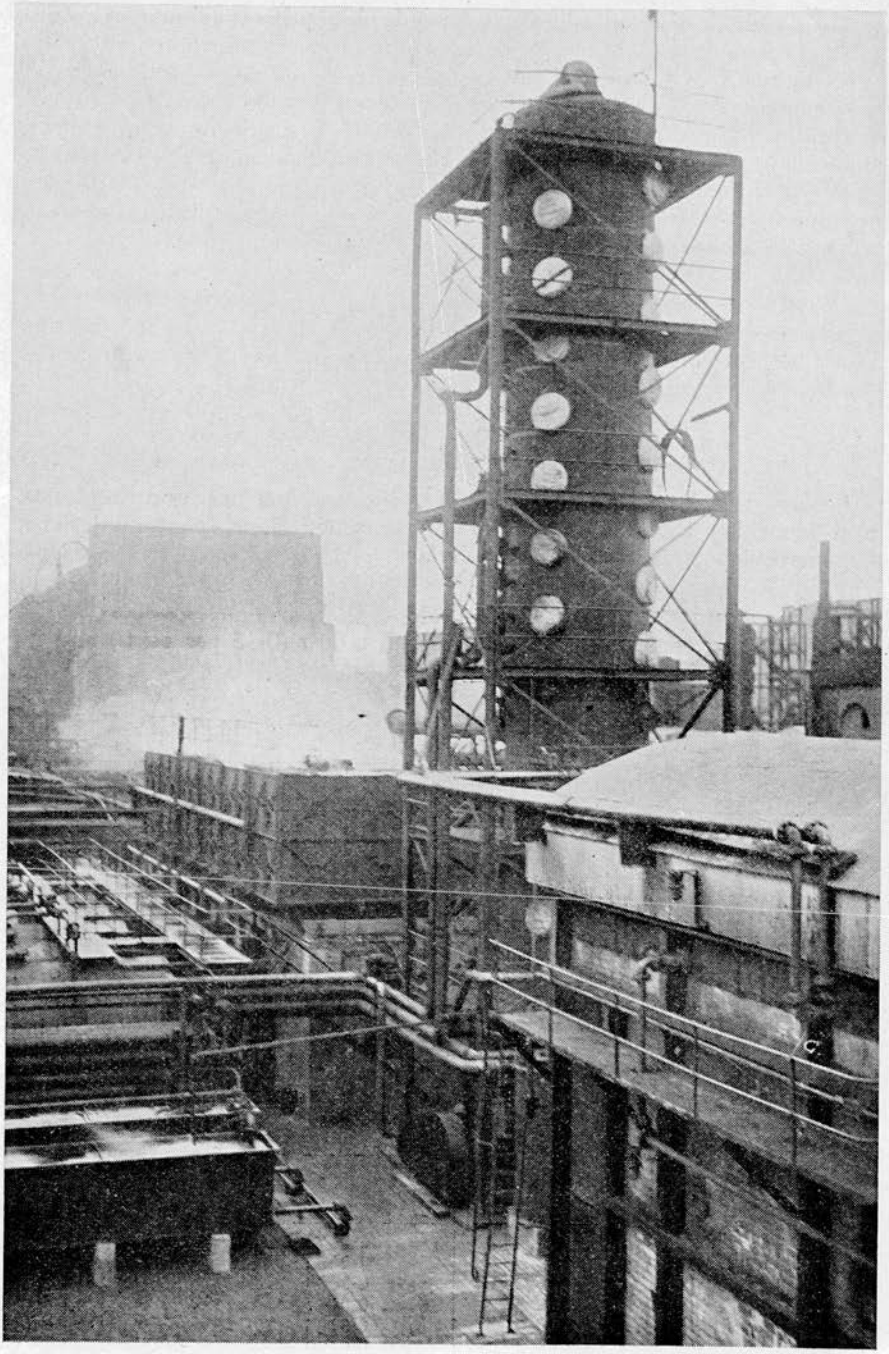
Distillation at 11 *mm. Hg.*

	Temp., ° C.	Sp. Gr.	Sulphur, %.
Fraction 0–20	46·5–128	0·8057	0·211
,, 20–40	128 –182·5	0·8522	0·297
,, 40–60	182·5–235	0·8777	0·307
,, 60–80	235 –285	0·8979	0·311
,, 80–90	285 –320	0·9101	0·348

The sulphur compounds present in the light fractions of Scotch shale oil are mercaptans, thioethers and thiophenes. Carbon disulphide is absent. No information is available as to the composition of the higher-boiling sulphur compounds. The extent to which the sulphur compounds in shale oil decomposed on heating is illustrated in Table II and Fig. 1. The

TABLE II.

Temp., ° F.	H_2S Formed, gms./100 ml. of oil.
300	0·0068
400	0·0082
500	0·0085
600	0·0167
700	0·0198
750	0·0400
800	0·0756



CRUDE OIL DISTILLATION UNIT.

crude oil was heated in an autoclave for 4 hours at temperatures ranging from 300 to 800° F., and the H_2S formed was determined.

At 500° F. the decomposition of the sulphur compounds becomes very rapid, until at 800° F. almost 20 per cent. of total sulphur in the oil is converted to hydrogen sulphide.

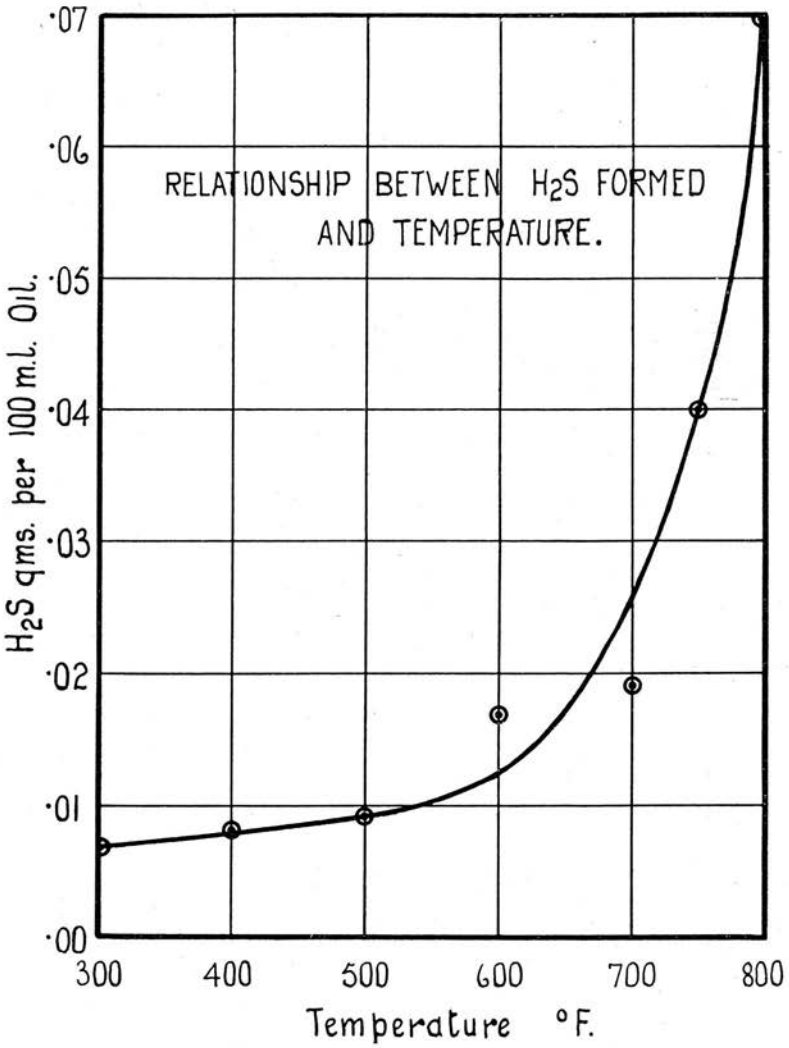


FIG. 1.

Nitrogen.

Nitrogen is present in the shale oil to the extent of 0·75 to 1 per cent. and exists mainly as basic compounds such as pyridine and its derivatives.

Robinson [6] and Robinson and Goodwin [7] identified the following bases of the leucoline series: $C_{12}H_{13}N$, $C_{13}H_{15}N$, $C_{14}H_{17}N$, $C_{16}H_{21}N$, whilst Garret and Smythe [8] isolated and identified pyridine, 2-methylpyridine,

2 : 6-dimethylpyridine, 2 : 4-dimethylpyridine, 2 : 5-dimethylpyridine, and 2 : 4 : 6-trimethylpyridine. Beilby [9] showed that of the total nitrogen present in the oil shale only 20–25 per cent. appears in the oil. On distilling the crude oil to coke, 50 per cent. of the nitrogen is lost as ammonia.

Phenols.

The phenol content of Scotch shale oil varies from 2 to 4 per cent. Gray [10] separated and identified phenol, *ortho*cresol, *meta*-cresol and xylenols, and showed the presence of guaiacol. Crude spirit from cracking shale oil contains about 1 per cent. of phenols, of which have been isolated cresols, xylenols and higher phenols, xylenols predominating.

II. Refining of Shale Oil.

It is possible to produce from Scotch shale oil a whole range of products—spirit, kerosene, gas oil, light lubricating oil, diesel oil, wax, fuel oil and coke. This process of " full refining " has been adequately described by Bailey,[3] and by Smith, Allen and Grant,[11] and while modern equipment and refining technique would probably alter the application of the methods described by these authors, the basic principles would be substantially the same. It is proposed to deal here with the manufacture of what are to-day the most important products of the shale industry—diesel oil and motor spirit,

Table III.

Analyses of Shale Oil and Scrubber Naphtha.

	Crude Oil.		Scrubber Naphtha.
Carbon	85·83%		—
Hydrogen	12·64%		—
Nitrogen	0·74%		—
Oxygen (by diff.)	0·33%		—
Sulphur	0·46%		—
Chlorine	Nil		—
Sp. gr.	0·877		0·736
Flash point	140° F.		—
Setting point	85 ,,		—
	Engler Dist.	Dist. at 10 mm. Hg.	Engler Dist.
I.B.P.	160° C.	48° C.	48° C.
10% dist. at	222 ,,	97 ,,	78 ,,
20% ,,	256 ,,	128 ,,	87 ,,
30% ,,	285 ,,	159 ,,	95 ,,
40% ,,	314 ,,	183 ,,	102 ,,
50% ,,	338 ,,	207 ,,	110 ,,
60% ,,	355 ,,	232 ,,	118 ,,
70% ,,	369 ,,	256 ,,	129 ,,
80% ,,	379 ,,	283 ,,	146 ,,
90% ,,	384 ,,	323 ,,	172 ,,
F.B.P.	384 ,,	346 ,,	245 ,,
Total distillate	96·5%	96·0%	98·0%
Residue	3·0%	2·5%	1·0%
Loss	0·5%	1·5%	1·0%

the term diesel oil being limited to fuel suitable for the modern high-speed compression-ignition engine. Two distinct processes are described: refining to (1) maximum diesel oil, which necessarily includes wax extraction and refining, and (2) maximum motor spirit. Shale oil from the present retorts is collected in two portions: (1) crude oil from the condensing system, and (2) "scrubber naphtha," the light spirit extracted from incondensable gas, and these two products are processed separately in the refinery.

Manufacture of Diesel Oil.

Scotch shale oil is an ideal base for diesel oil, and yields on appropriate treatment 40 to 50 per cent. of first-grade light diesel oil.

Alternative refining reagents which have been considered are sulphuric acid and liquid SO_2. Whilst there are possibilities of using the above reagents in one particular refining scheme, each in its most useful sphere, they are dealt with separately here.

Refining by Sulphuric Acid (sp. gr. 1·84).

In refining with sulphuric acid the main operations are:—

(*a*) Distillation to coke.
(*b*) Extraction of wax from press cut.
(*c*) Acid and soda treatment of wax-free oil.
(*d*) Re-running treated diesel oil.
(*e*) Pressure distillation of the residue from (*d*).

(*a*) *Distillation to Coke.*

The carbon/hydrogen ratio of shale oil is higher than that of the average petroleum, and at the same time the oil is more unstable, so that a distillation to coke effects a mild cracking, yielding a distillate more saturated than the crude oil. This distillation, therefore, is a definite step in refining as well as a means of fractionating the crude. Consideration has been given as to whether distillation under vacuum or under normal pressure would be most advantageous, and a few of the major points brought out during this inquiry are given below.

The work was carried out in a 5-gallon still with a short fractionating column at pressures varying from 15 mm. Hg to normal pressure. In all cases the distillate was cut to spirit, wax-free cut, pressible cut and residue. A 10 per cent. residue was found to contain all the amorphous wax, and was then distilled to coke at atmospheric pressure under mild cracking conditions, producing a distillate containing crystalline wax, which was added to the pressible cut. The pressible cut was dewaxed and blended with the wax-free cut, and the blend treated with sulphuric acid and caustic soda and re-run to diesel oil. All treatments were identical, to give a true comparison, and diesel oil was refined to the same volatility in each case. The yields of three typical tests and essential characteristics of the diesel oil are given below:—

TABLE IV.

Diesel Oil.	Distillation Pressure.		
	15 mm. Hg.	330 mm. Hg.	Atmospheric.
Loss on treatment, % on crude . . .	10·6	10·6	10·0
Yield, % on crude	53·5	59·9	61·3
Aniline point, ° C.	55	57	58
Carbon residue (Conradson), % . . .	0·10	0·03	0·008
Viscosity (Redwood No. 1) at 100° F., secs..	38	35	35

Distillation at atmospheric pressure therefore gives not only a higher yield of diesel oil, but also a better diesel oil, as indicated by the higher aniline point and lower carbon residue. The effect of the distillation at atmospheric pressure is to decompose unsaturated polymers which are to a certain extent preserved in vacuum distillation.

In the refinery the distillation of the crude oil to a 15 per cent. residue is effected in a pipe-still and bubble-tower unit of conventional design (see flow diagram, Fig. 2). The products taken are :—

TABLE V.

Overhead	Crude spirit	6%	Sp. gr. 0·778
Tray cut	Wax-free cut	23%	,, 0·816
,, ...	Pressible cut	56%	,, 0·890
Residue		15%	,, 0·960

The feed temperature necessary to run to a 15 per cent. residue is 750–760° F. when operating at 1 lb. of steam per gallon of distillate. It is possible to work to 10 per cent. residue, but the free carbon content of the residue, due to increased decomposition of the oil, is rather high, whereby the lower trays of the fractionating column are liable to be fouled. The residue contains all the amorphous wax cut which is in the fraction 10–2 per cent. on crude. By coking this residue under mild cracking conditions, the amorphous wax is rendered crystalline and at the same time the residue is partly cracked to lighter oils. The distillate from the coking stills is thereafter recycled as feed to the crude unit. The products from this distillation and coking are : spirit, wax-free cut, pressible cut and coke.

The coking operation is carried out batch-wise in pot stills. The coke is valuable on account of its low volatility (5–7 per cent.) and ash content (less than 0·5 per cent.) for making carbon electrodes for the aluminium industry. In some cases a coke of even lower volatility is required, but it is impossible to produce this in the conventional coking stills. The volatile matter content can be reduced to 1 per cent. by processing the coke through an ordinary Scotch shale retort operating at coke temperatures of 1400° F. The optimum throughput is 3 tons per retort per day, and is best carried out in a current of steam. Under these conditions the loss is 12 per cent. To reduce the volatile matter still further, some type of calciner with direct heating is necessary, such as that described by Watkins.[12]

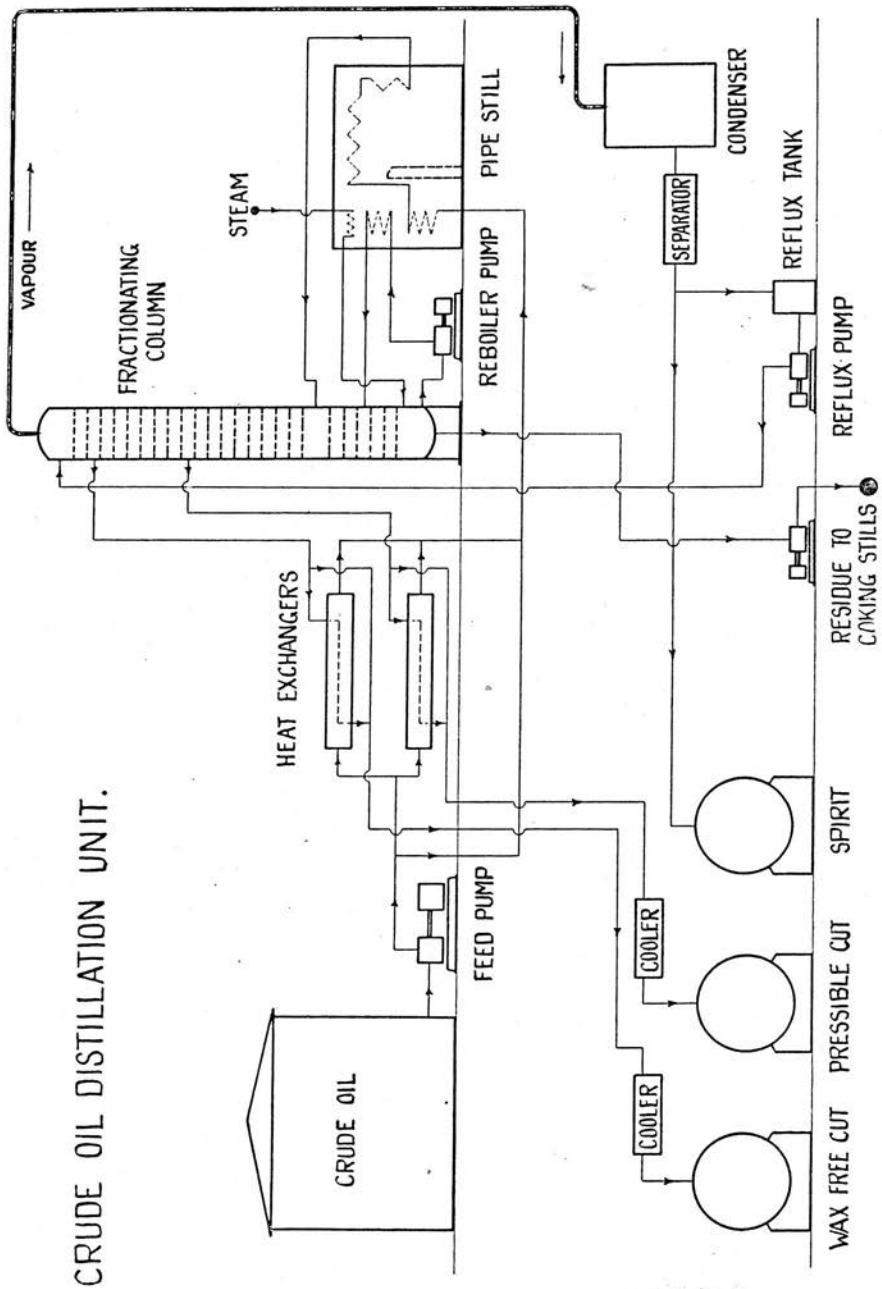

CRUDE OIL DISTILLATION UNIT.
VAPOUR
FRACTIONATING COLUMN
STEAM
PIPE STILL
REBOILER PUMP
CONDENSER
SEPARATOR
REFLUX TANK
REFLUX PUMP
RESIDUE TO COKING STILLS
HEAT EXCHANGERS
FEED PUMP
COOLER
COOLER
CRUDE OIL
SPIRIT
PRESSIBLE CUT
WAX FREE CUT

FIG. 2.

Below are given analyses of the normal production of shale coke (5–7 per cent. volatile matter) and coke of volatile matter 1 per cent.

TABLE VI.

	Normal Shale Oil Coke.	Low Volatility Shale Oil Coke.
Moisture	1·84%	1·92%
Volatile matter (less moisture)	5·28%	1·00%
Fixed carbon	92·50%	96·64%
Ash	0·38%	0·44%
Carbon	92·50%	94·60%
Hydrogen	2·40%	0·60%
Sulphur	0·38%	0·40%
Calorific value (B.Th.U./lb. (gross)	14,800	13,980

(*b*) *Extraction of Wax from Press Cut.*

The pressible cut itself is of too high viscosity to ensure satisfactory filtration, and is blended back with wax-free cut to a viscosity of 50 secs. (Redwood No. 1) at 100° F. The wax is extracted in two stages, leaving a dewaxed oil, setting point 22° F. The extraction and refining of the paraffin wax are dealt with in a separate section later.

(*c*) *Acid and Soda Treatment of Wax-free Oil.*

The dewaxed oil blended with the wax-free cut and spirit still residue is termed " Crude Diesel Oil," and is subjected to treatment with sulphuric acid, sp. gr. 1·84, and caustic soda, 10 per cent. solution.

The possible variations in acid treatment are :—

(i) Divided treatment—*i.e.*, treatment in various fractions, giving each fraction the appropriate treatment as against treatment of the diesel oil as a whole.

(ii) Percentage treatment.

(iii) Temperature of treatment.

(i) *Divided Treatment.*

The crude shale oil was cut into four fractions, viz. 0–20 per cent., 20–30 per cent., 30–55 per cent., and 55–90 per cent. each fraction (after

TABLE VII.

Fraction.	% on Crude.	Sulphuric Acid Treatment, % on fraction.	Loss on Acid and Soda Treatment, % on fraction.	Treated Oil, % on crude.
0–20·2%	20·2	2·0	6·8	18·8
20·2–30%	9·8	2·0	9·8	8·8
30–55·1% (after dewaxing)	23·8	3·0	16·5	19·9
55·1–93·7% (after dewaxing)	33·6	3·0	18·5	27·4
Totals for divided treatment	87·4			74·9
Blend of all fractions	87·4	2·5	13·4	75·7

dewaxing the cuts containing wax) given the necessary treatments to give satisfactory diesel oils, and the results were compared with a straightforward treatment of the blended distillates. Below are given details of yields, and these figures show that no advantage is to be derived from treating the oil in separate fractions as compared with treating the oil as a whole.

(ii) *Percentage Treatment.*

Two series of tests were made: one in which acid alone was used, and the other where, before acid treatment, the oil was subjected to tar from a previous treatment. Results are given in Table VIII.

TABLE VIII.

Treatment Temperature, 65° F.

	Acid Alone.			Tar and Acid.		
Sulphuric acid, % by volume .	3·5	3·0	2·5	2·5	2·0	1·5
Finished Diesel Oil.						
Colour, ½-in. cell, Lovibond, 510 and 200 series	5·2Y 0·2R	5·0Y 0·4R	6·0Y 0·35R	5·2Y 0·1R	6·5Y 0·4R	15Y 0·85R
Stability test	Stable.	Stable.	Failed.	Stable.	Stable.	Stable.

The use of tar from a previous treatment shows a definite saving in acid. With acid alone 3 per cent. is necessary, but with tar only 2 per cent. is required to give the same product.

(iii) *Temperature of Treatment.*

The temperature range explored was 45–100° F. and the treatment in all cases was 3 per cent. of acid by volume. From Table IX, which shows the effect of increasing the temperature, it is concluded that the optimum temperature as regards colour is 65° F. It is noteworthy that the treatment at the highest temperature, 100° F., does not result in a greater loss than that at 65° F., and in actual practice the higher temperature is preferred, owing to the greater fluidity of the tar.

TABLE IX.

Sulphuric Acid Treatment—3% by Volume.

Initial Temperature of Treatment.	45° F.	65° F.	80° F.	100° F.
Loss on treatment, % by vol. .	13·2	15·1	14·6	14·8
Finished Diesel Oil.				
Colour, ½-in. cell . . .	9·5Y 0·55R	5·0Y 0·4R	5·0Y 0·4R	6·0Y 0·3R
Stability test	Failed.	Stable.	Stable.	Stable.

(*d*) *Re-running of Treated Diesel Oil.*

The treated diesel oil shows only 75–80 per cent. distilling to 350° C., and in addition certain polymerized products are formed during the acid treatment, so that the treated oil has necessarily to be re-run.

The question as to whether vacuum distillation or atmospheric distillation of this oil would be most advantageous was the subject of a separate inquiry, and the results showed that the diesel oil from vacuum distillation was slightly better in colour. Whilst this was not apparent in an oil of volatility 95 per cent. at 350° C., it was appreciable if the diesel oil was run to a high end-point such as 80 per cent. at 350° C., but the advantages of vacuum distillation were so small as to be disregarded.

Re-running is normally effected in a pipe-still and bubble-tower unit with steam at 1 lb. per gallon. A spirit cut is taken as overhead, diesel oil as tray cut, leaving a residue of 20 per cent.

(*e*) *Pressure Distillation of Residues.*

This 20 per cent. residue is still a potential source of diesel oil, and can be converted into diesel oil and spirit by pressure distillation at 50–75 lb. per sq. in. and 780–790° F., yielding approximately 30 per cent. of finished spirit, 40 per cent. finished diesel oil, pitch 15 per cent., gas and loss 15 per cent. The diesel oil from cracking this material, unlike cracked gas oil from petroleum, which shows considerably lower aniline point than straight-run gas oil from the same source, is of approximately the same aniline point as diesel oil from the straight distillation of the shale crude—*i.e.*, aniline point 63° C. The diesel oil thus produced requires less acid to refine, and the losses are correspondingly lower.

Another potential source of diesel oil is wax. On pressure distillation, 50 lb. per sq. in., the wax yields spirit 35 per cent. diesel oil 45 per cent. coke and gas 20 per cent. The diesel oil fraction, however, whilst of high aniline point (76° C.) is of very low specific gravity (0·785) and viscosity (34 secs. at 70° F.), which at the moment are considered disadvantages. As expected, the spirit produced is of poor engine test (Octane No. 39), and to get this to anything like a commercial octane rating would mean a very high loss in reforming.

Refining Scheme.

A typical scheme for the production of diesel oil from shale oil with sulphuric refining acid is given on the opposite page.

Properties of Shale Diesel Oil.

Shale diesel oil manufactured by refining with sulphuric acid and caustic soda has the following characteristics. (Table X.)

Refining by SO_2 *Treatment.*

The natural alternative to sulphuric-acid refining is extraction by liquid SO_2. This has been applied experimentally to crude shale-oil distillate both before and after dewaxing. The treatment of the wax-bearing distillate offered several advantages, the principal of which was the production of a crystalline and cleaner wax, but, on the other hand, the treatment has to be carried out in two stages : one at a comparatively

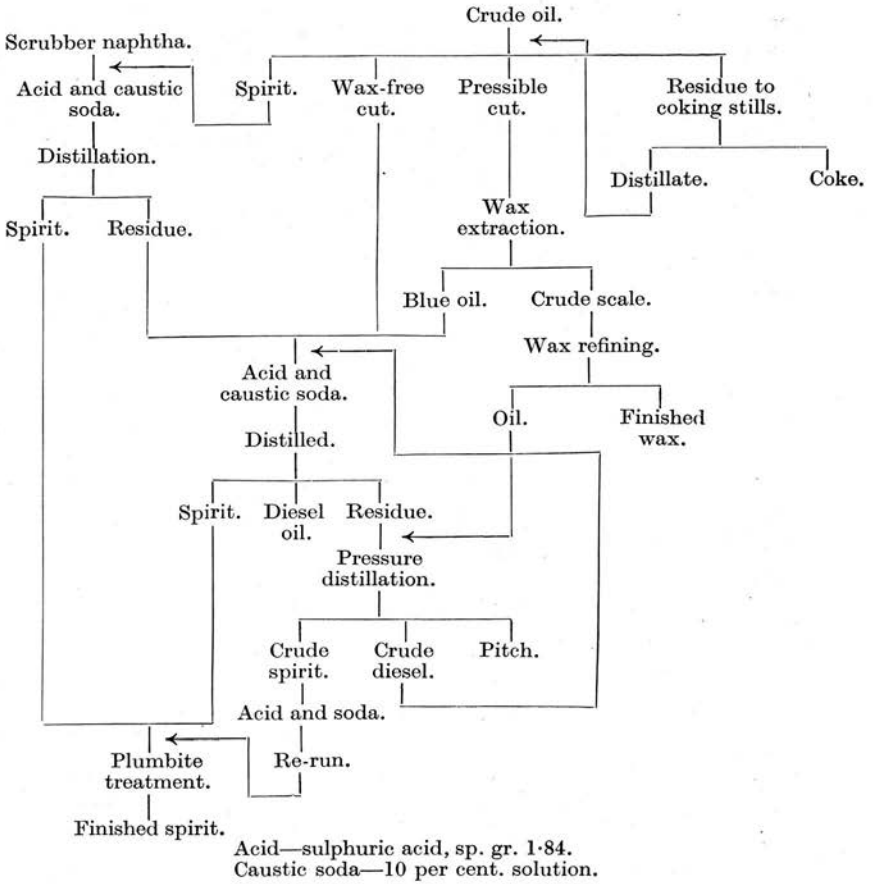
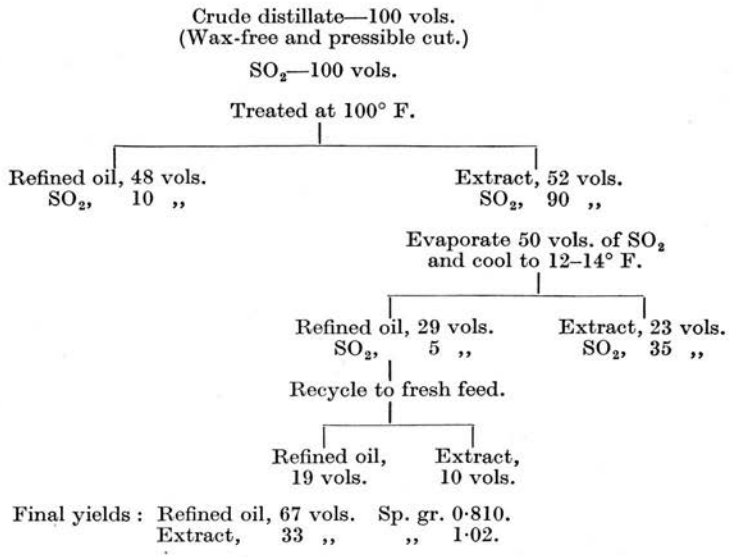


TABLE X.

Analysis.

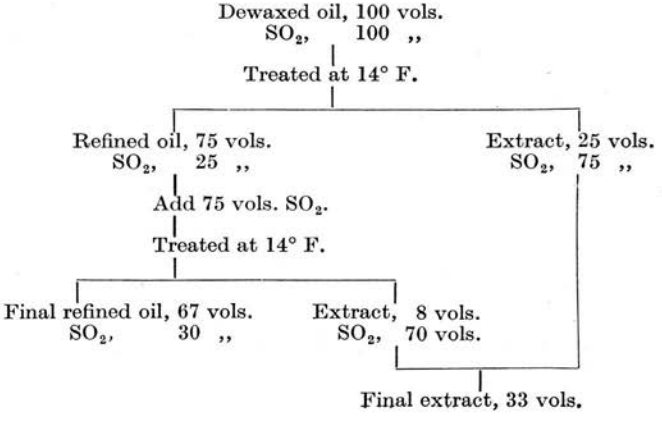
Sp. gr	0·841
Aniline point	64° C.
Sulphur	0·32%
Carbon residue (Conradson)	Nil.
Cal. val., B.Th.U./lb. (gross)	19,530
Setting point (I.P.T.)	12° F.
Cetene number	72
I.B.P.	209° C.
10% dist. at	233 ,,
20% ,,	244 ,,
30% ,,	258 ,,
40% ,,	270 ,,
50% ,,	284 ,,
60% ,,	300 ,,
70% ,,	316 ,,
80% ,,	330 ,,
90% ,,	350 ,,
F.B.P.	374 ,,
Total distillate	98·5%
Residue	1·0%
Loss	0·5%

high temperature, which results in a somewhat complicated operation. Such a scheme may be represented by the following flow diagram.



The refined oil is dewaxed, given a light sulphuric acid treatment, and re-run to yield diesel oil. The extract is a non-waxy, low-setting-point, viscous oil, suitable for blending into fuel or furnace oils.

The application of liquid SO_2 to dewaxed oil is a perfectly straightforward procedure. The optimum temperature of treatment is about 14° F. In one typical experiment the oil was treated with liquid SO_2 (200 per cent. by vol.) in Edeleanu's experimental apparatus in two stages, with the results shown below. A two-stage treatment of kerosene in this apparatus corresponds to the normal countercurrent treatment of this oil in the commercial unit.



The diesel oil obtained from the refined oil by neutralizing with caustic soda and distilling was not to specification as regards colour, and a light acid treatment (½ per cent. of 96 per cent. acid followed with caustic soda) was necessary before distillation. Table XI gives particulars of crude dewaxed oil, extract and finished diesel oil.

TABLE XI.

	Dewaxed Oil before SO_2 Treatment.	Refined Oil.	Extract.	Finished Diesel Oil.
C	85·98%	—	86·32%	—
H	12·23%	—	9·70%	—
N	0·50%	—	1·22%	—
S	0·34%	0·18%	0·94%	0·14%
O (by diff.)	0·95%	—	1·82%	—
Sp. gr.	0·878	0·829	0·988	0·817
Flash point	145° F.	179° F.	185° F.	171° F.
Viscosity (Redwood No. 1):				
At 70° F.	51 secs.	44 secs.	152 secs.	38 secs.
,, 100 ,,	42 ,,	38 ,,	70 ,,	35 ,,
Aniline point	57·4° C.	80·4° C.	Less than 0	78° C.
Setting point	20° F.	28·5° F.	,,	22° F.
Carbon residue	0·27%	0·06%	2·04%	Nil.
Bromine value	14	19	10	—
Hard asphalt	0·03%	Nil.	0·06%	—
Cal. val. (B.Th.U./lb. gross)	19,010	19,710	17,660	19,920
Colour (½-in. cell)	—	—	—	5·5Y + 0·3R
I.B.P.	192° C.	199° C.	190° C.	199° C.
10% dist. at	233 ,,	232 ,,	233 ,,	228 ,,
20% ,,	255 ,,	253 ,,	260 ,,	245½ ,,
30% ,,	275 ,,	274 ,,	288 ,,	261 ,,
40% ,,	296 ,,	294 ,,	308 ,,	275 ,,
50% ,,	314 ,,	310 ,,	326 ,,	290½ ,,
60% ,,	331 ,,	327 ,,	340 ,,	303 ,,
70% ,,	—	344 ,,	—	316 ,,
80% ,,	—	—	—	331 ,,
90% ,,	—	—	—	350 ,,
F.B.P.	>350 ,,	>350 ,,	>350 ,,	>350 ,,
Total distillate	70·0%	75·0%	64·0%	92·0%
Residue	30·0%	25·0%	36·0%	7·5%
Loss	Nil.	Nil.	Nil.	0·5%
% dist. at 200° C.	—	—	1·5	—
,, 300 ,,	42·5	43·5	36·0	58·0
,, 350 ,,	69·0	73·0	63·0	90·0

The extract as such was only of fuel value, and two methods of dealing with it have been investigated—viz., (1) cracking to spirit, and (2) hydrogenating to diesel oil.

(1) It is possible to crack the extract to spirit, but the spirit yields are low. In one series of tests in the laboratory autoclave, cracking to coke, the extract gave :—

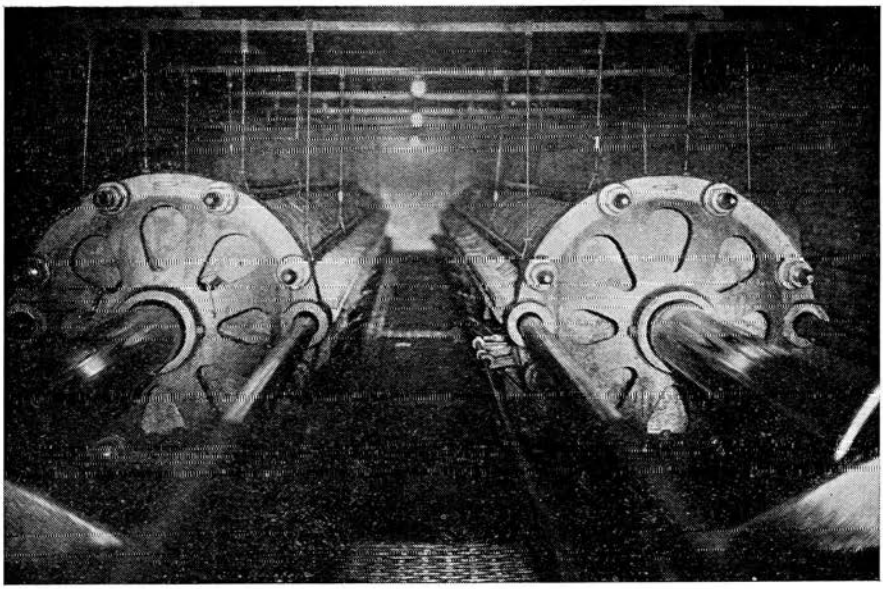
Spirit	30%
Coke	45%
Loss and gas	25%

The crude spirit, after refining with sulphuric acid and caustic soda and re-running to F.B.P. 200° C., was of specific gravity 0·783 and Octane No. 59, which is only slightly higher than normal cracked spirit from shale

oil. The sulphur content of the spirit was high, and difficulties were encountered in refining the oil to a suitable colour and stability. The loss on treatment amounted to 7·5 per cent.

(2) The possibility of hydrogenating the extract to give diesel oil seemed practicable, but so far, while the yields—as would be expected—have been good, the cetene value of the oil is low.

The extract, therefore, has proved to be of fuel value only, and at the present time sulphuric-acid treatment, including the recovery of the residual acid which is used for the manufacture of sulphate of ammonia, is the more attractive. Special burners have been designed to deal with the washed and neutralized acid tar, so that the material extracted by the acid is still available as works fuel, the calorific value being 15,500 B.Th.U. per lb.



WAX FILTER PRESS.

III. Wax Extraction and Refining.

In any method of refining shale oil to diesel oil, the other products incidentally produced are motor spirit, wax and fuel oil. The motor spirit is derived from three sources : (1) spirit extracted from the retort gases by absorption with gas oil, (2) spirit in crude oil, and (3) spirit produced by cracking the residue distilling above the diesel-oil range. The refining of this spirit is dealt with later when refining to maximum spirit is discussed.

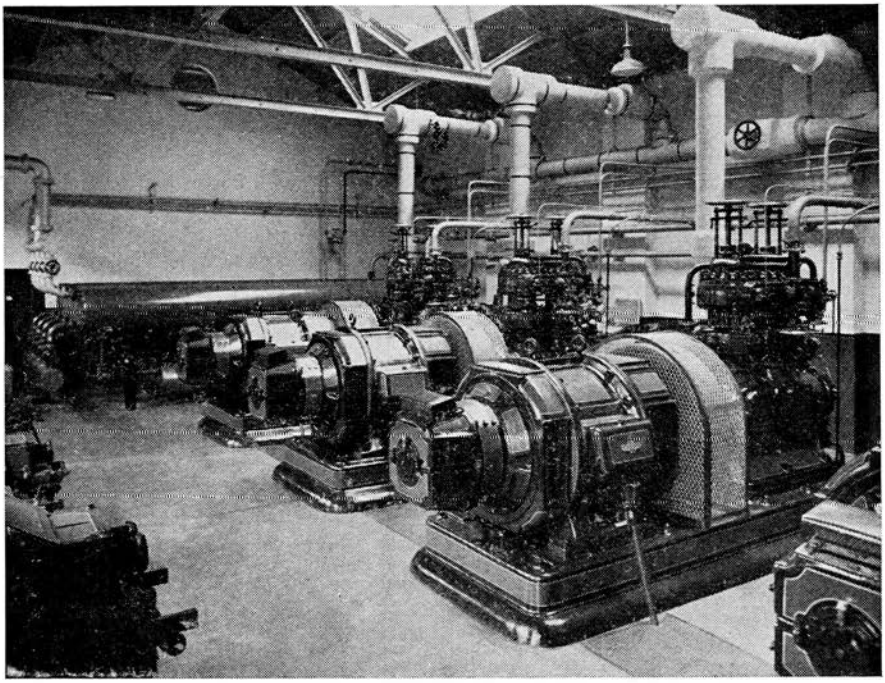
The extraction and refining of wax may be conveniently divided into the following sections :—

(*a*) Extraction.
(*b*) Treatment.
(*c*) Sweating.
(*d*) Decolorizing.
(*e*) Finished Waxes.

(*a*) *Extraction.*

The pressible fraction, of specific gravity 0·880 at 60° F., setting point 80° F., viscosity 50 secs. at 100° F. and containing 18 per cent. wax, is cooled and filtered in two stages, the scales obtained being refined separately.

The pressible cut is first cooled to 40° F. and the wax which separates filtered off. The blue oil is then cooled to 22–23° F. and again filtered. The first filtration is carried out at 40° F., because at that temperature a scale is obtained which, when sweated to the highest-melting-point wax prepared, gives a wax of the desired oil content. Under these conditions



AMMONIA COMPRESSOR HOUSE.

also, the second scale is suitable for the preparation of low-melting-point waxes. The temperature of 22–23° F. for the second filtration was chosen to give a blue oil which, when blended with the wax-free cut and redistilled, gives the necessary setting point and cloud point in the finished diesel oil. If the temperature of the second cooling is allowed to fall two or three degrees below that given, a considerable increase in the oil content of the scale is noticed.

The plant used for dewaxing at each stage consists of a heat exchanger, two coolers and two filters, with the necessary pumps and ammonia compressors. In the heat exchanger the oil is cooled by means of cold filtered oil, and in the coolers the cooling medium is liquid ammonia. These

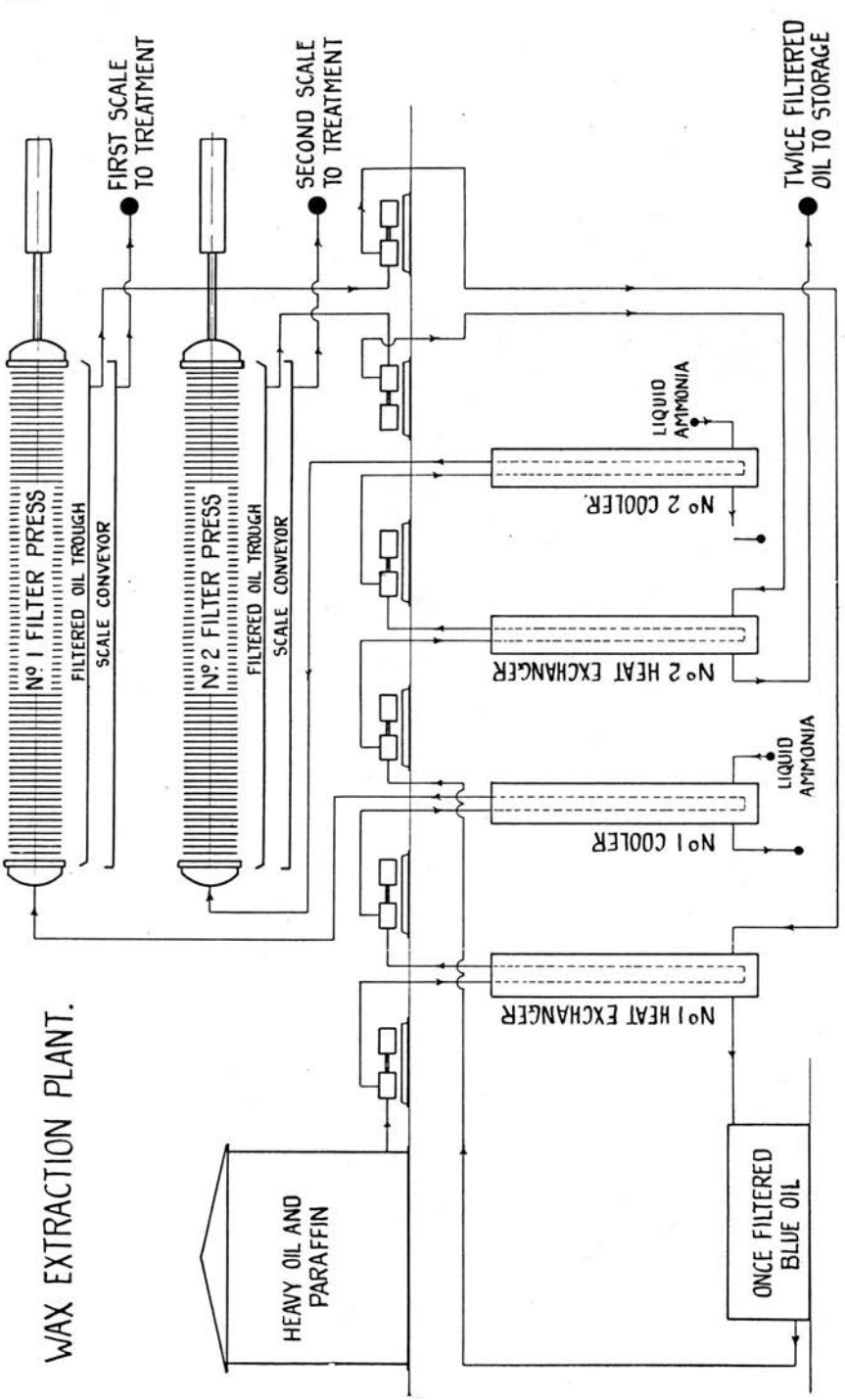

WAX EXTRACTION PLANT.
Nº 1 FILTER PRESS
FILTERED OIL TROUGH
SCALE CONVEYOR
Nº 2 FILTER PRESS
FILTERED OIL TROUGH
SCALE CONVEYOR
FIRST SCALE TO TREATMENT
SECOND SCALE TO TREATMENT
TWICE FILTERED OIL TO STORAGE
LIQUID AMMONIA
Nº 2 COOLER
Nº 2 HEAT EXCHANGER
LIQUID AMMONIA
Nº 1 COOLER
Nº 1 HEAT EXCHANGER
HEAVY OIL AND PARAFFIN
ONCE FILTERED BLUE OIL

FIG. 3.

coolers are kept full of liquid ammonia by means of a constant-level device, the gaseous ammonia produced during the cooling being drawn off by the compressor, compressed, cooled and returned to the cooler. The filter presses are of the horizontal hydraulic-plate type with loose rings. There are 510 plates in a press, and these are 3 ft. 9 in. in diameter and the rings $\frac{1}{2}$ in. thick (see flow diagram, Fig. 3).

The pressible cut is pumped through the heat exchanger and cooler into the filter. The pressure on the filter is not allowed to exceed 450 lb. per sq. in., this pressure corresponding to a pressure of 600 lb. per sq. in. at the pump. For maximum oil removal from the scale it has been proved that the pressure should be as high as possible and should be maintained as long as possible. Under the present method of operating, the rate of filtration, including time taken for cleaning, works out at 0·128 gal. per sq. ft. per hr.

One of the major drawbacks to this type of filterpress is buckling of the plates. The plates bend in different directions, and so give thick and thin cakes of scale, and sometimes empty spaces are left. When a filter is in this state, the results are unsatisfactory, the filter frequently bursts before the pumping time is complete and the thick cakes of scale contain two to three times the oil of a normal-sized cake. This trouble has been largely got over by regularly turning bent plates so that they all face in the one direction. This operation is performed once a week.

In the second-stage filtration—that is, when filtering at 22–23° F.—it is found that the rate of filtration gradually becomes slower and the scale obtained becomes sticky and oily, due to the pores of the cloths clogging up; and to overcome this, it is necessary to wash the filter by injecting hot oil.

The importance of obtaining a clean, well-fractionated press cut cannot be over-stressed, as the presence of relatively small quantities of high-boiling-point asphaltic material results in a black, sticky paste being deposited in the filters from which oil cannot be pressed and which is very difficult to remove from the filter.

Under normal operating conditions a first-stage scale of melting point 118° F. and expressible oil content 15 per cent. is obtained, whilst the second scale has a melting point of about 97° F. and oil content of 25 per cent. The total scale extracted amounts to 22 per cent. of the pressible fraction, the ratio of first scale to second scale being 3 to 1. The twice-cooled blue oil has a specific gravity of 0·890, viscosity 70 secs. at 70° F., and setting point 22° F.

(*b*) *Treatment.*

The crude scales obtained from the filter presses are dark in colour and unstable, and are treated with sulphuric acid to give as low a colour as possible before being sweated.

The scales are settled until free from water and sludge, and then treated with sulphuric acid of specific gravity 1·84. This treatment is carried out in a vertical cylindrical vessel, the agitation being by air blowing. The acid treatment is given in two stages, a small quantity first to remove any moisture, followed, after tar removal, by the bulk of the acid. The quantity

of acid necessary varies with the oil content of the scale, and normally the quantity required is about 2 per cent. for scale from first cooling and 2·5 per cent. for scale from second cooling.

In order to obtain the lowest possible colour in the treated scales, the points to be watched are : (1) quantity of acid used—insufficient acid or excess acid should be avoided ; (2) the temperature during treatment should be as low as possible ; (3) scale should not be left in contact with acid any longer than is necessary for settling of the acid tar.

The acid-treated scales are neutralized by agitation with 10 per cent. sodium carbonate solution, this being used in preference to caustic soda solution because considerably lower colours in the treated scales are obtained and sodium carbonate is less liable to give troublesome emulsions than caustic soda. The neutralized scales are next washed two or three times with hot water to remove traces of alkali. This water-washing is important, since scales containing alkali give trouble in the sweating process. The scale does not cool to give a hard cake, but is softer than usual, and oil removal is difficult. The effect is seen both in the strainings and in the wax left in the pans.

The average colours of treated scales are—first scale, $9Y + 1R$, and second scale, $12Y + 2R$, determined in a $\frac{1}{2}$-in. cell, and since these products are not perfectly colour stable, they are stored at as low a temperature as possible and for as short a period as possible before being sweated. The loss in treatment with acid and sodium carbonate amounts to between 2 and 3 per cent.

(*c*) *Sweating.*

The treated scales are next sweated to remove oil and to divide the waxes into various grades according to melting points. The plant consists of twenty-four Henderson houses (tray type) and one Alanmor stove, the Henderson houses being of approximately 17 tons capacity and the Alanmor stove of 20 tons.

From scale of melting point 118° F. are prepared waxes ranging in melting point from 118–120° to 125–130° F., whilst waxes ranging from 100–105° to 110–115° F. are prepared from the scale of melting point 97° F. The melting point and oil content of the two scales are such that when these have been sweated to the highest-melting-point wax prepared from that grade, the oil content of the wax is at the desired figure. The lower-melting-point waxes in each case are then prepared from sweatings.

The first fraction obtained from every sweating consists of oil with wax dissolved in it, and all fractions with melting point below 90° F. are collected together. The bulk, of melting point 85° F., oil content 60 per cent., and amounting to 30 per cent. on scales charged for sweating, is transferred to cracking stock. A second fraction is collected from every sweating, and these consist of wax mixtures of various melting points and oil contents. These fractions are blended with fractions which give like melting points for the oil-free waxes contained in them, five different grades of sweatings being collected. These fractions are re-sweated separately to give different grades of waxes ; the higher the melting point of the oil-free wax contained in a fraction, the higher the melting point of the wax prepared from it,

care being taken in every case that there is a sufficient gap between the melting point of the oil-free wax in the fraction and the melting point of wax to which it has to be sweated, to ensure that the sweating will be of such a duration that when the melting point desired in the wax is reached, the oil content will also be at the desired figure. This method of blending sweatings gives more satisfactory results than blending on the basis either of oil content or melting point.

In practice a number of schemes are possible, but the scheme adopted should be so arranged that adjustments can be made to suit the requirements for various grades, with the minimum change in composition of the products produced. A typical scheme of operating is given, whilst Table XII gives analyses of different fractions on which this scheme is based.

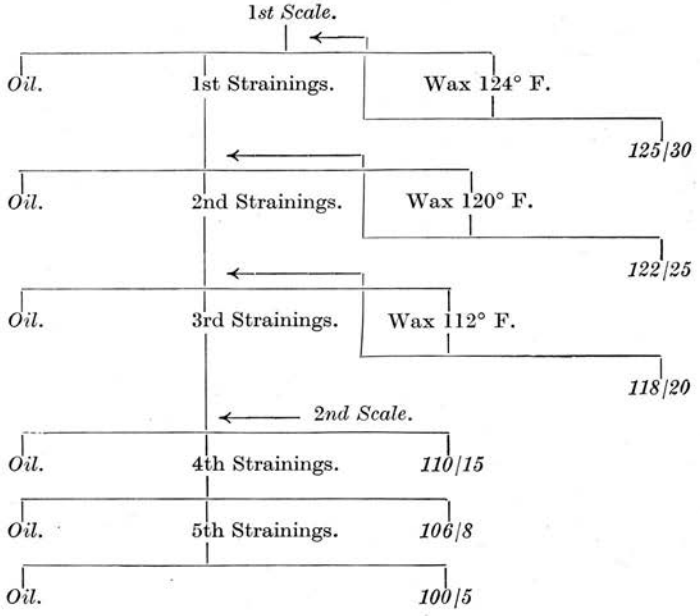


TABLE XII.

Analyses of Scales and Strainings.

	Melting Point, ° F.	Oil Content, %.	Melting Point of Oil-free Scale, ° F.
1st Scale	118	15	122
1st Strainings	108	20	113
2nd ,, ...	104	20	109
3rd ,, ...	98	15	102
2nd Scale	97	25	103
4th Strainings	96	15	100
5th ,, ...	93	20	98

The stoves are charged with scale at such a temperature that solidification does not take place immediately, and are allowed to cool. During the sweating the temperature is raised very slowly at the beginning and up to the point where fractions of melting point below 90° F. are collected. The temperature is then quickly raised to a fixed maximum, depending on the fraction being sweated, and is maintained at that figure until the sweating is finished. This slow sweating at the beginning gives better separation between oil and wax, and so reduces the amount of re-sweating to be done.

When the yield of wax from any particular sweating is low, double sweating is resorted to. By this means the quantity of wax per house is increased and at the same time a better product is obtained. The amount of sweating and re-sweating carried out can be judged from the fact that for every ton of finished wax produced, $4\frac{1}{2}$ tons of scale or sweatings are dealt with.

To obtain uniform products throughout the range, it is important that the two scales charged to the sweating plant should be of constant composition and the different grades of waxes should then always be prepared in the same way.

(*d*) *Decolorizing.*

The waxes from the sweating plant are still too high in colour to be despatched without further treatment. The colour is removed and the different grades are prepared to colour specification by running the melted waxes through previously ignited Florida earth of 16–30 mesh per inch, contained in tall vertical columns. The wax to be treated is settled until free from moisture, and then filtered through the Florida earth at a definite rate, the temperature being kept as low as possible. The colour of the filtered wax is very low at the start of the run and gradually rises, the filtration being stopped when the bulk of filtered wax is of the necessary colour.

The filters are allowed to drain and the Florida earth is then washed free from wax by circulating naphtha through the filters. The naphtha is then removed by steaming, and the Florida earth reactivated by heating in an oil-burning rotary furnace at a temperature of 900° F. The mixture of naphtha and wax is separated by steam distillation, the naphtha being used again and the wax returned for re-sweating. The quantity of wax returned amounts to 20 per cent. of the wax decolorized.

The quantity of Florida earth used for decolorizing is about half a ton for every ton of decolorized wax obtained, whilst the fresh earth necessary as make up for mechanical loss amounts to 0·04 of a ton per ton of wax.

(*e*) *Finished Waxes.*

The molten wax is cast into trays of capacity 14 lb. and allowed to cool at atmospheric temperature, care being taken that the temperature before casting is not too low, otherwise rough-surfaced cakes are obtained due to the wax solidifying too quickly.

The waxes of melting point above 118° F. are comparatively transparent, whilst those of lower melting point are more or less opaque. All grades, however, are more transparent than corresponding grades prepared by rapid

cooling, as in press moulding. Opaqueness and mottling in waxes cast at atmospheric temperature have been found to be due to the presence of air and oil and the removal of either results in transparent wax, free from mottling.

All grades are run to a definite melting point and colour, but to meet the specification for certain grades, special tests have to be applied. These include loss on heating at a given temperature, penetration and plasticity tests. Over and above these tests, however, all waxes are examined daily in the Jackson–Burmah testing machine which determines oil content and width of cut.

IV. Refining to Maximum Spirit.

(*a*) *Straight-run Spirit.*

The oil products from the retorting of shale consist of scrubber naphtha and crude oil in the ratio of 15 to 85. Of the former 95 per cent. is within the motor-spirit range, and of the latter 5 per cent. giving a total yield of spirit existing as such in the scrubber naphtha and crude oil of 18·5 per cent. In refining, the crude oil is topped and the distillate to 200° C. is added to the scrubber naphtha and the blend refined as a whole. The refining of this material to specification presents no special difficulties. The crude spirit is treated with sulphuric acid (sp. gr. 1·84) and caustic soda in a pump orifice washer and re-run in a pipe-still unit, the distillate being finished by plumbite treatment.

As well as motor spirit, various grades of solvents are prepared from this same base stock. These solvents are used in the rubber, linoleum and cleaning industries, and are excellent solvents due to their comparatively high proportion of unsaturated compounds.

(*b*) *Cracking.*

The residue distilling above 200° C. can be converted into motor spirit either by cracking or hydrogenation. Both methods have been investigated experimentally, but the former has been general commercial practice during the past nine years.

Shale oil is comparatively easily cracked, and when working to maximum spirit production low-pressure liquid phase operation gives satisfactory results. At 795–800° F. and 175 lb. per sq. in. pressure, crude shale oil and shale-gas oil yield respectively approximately 55 and 65 per cent. spirit. As would be expected, however, the spirit under these cracking conditions is of low Octane No. Working at higher temperatures, 1000–1100° F., either under pressure or on vapour-phase operation, gas oil and kerosene give lower yields of higher Octane No. spirit. In one test, kerosene and pressure distillate bottoms from the liquid phase plant were cracked in a vapour-phase unit at 1100° F. and gave the following yields :—

Spirit	52% by volume.
Fuel oil	10% ,,
Gas, loss and coke . . .	38% ,,

In this case the Octane No. of the spirit was 70, but, as seen, the yield was very low compared with the low-pressure cracking of this stock, which

yielded 70–75 per cent. spirit. This yield of spirit could be raised to approximately 60 per cent. by increasing the pressure to 800–1000 lb. per sq. in.

While in the existing units only crude oil and gas oil have been processed individually, the relative yields and ease of cracking of various cuts from shale oil have been determined in the laboratory autoclave, and these yields by the use of known factors can be converted into commercial yields. In this series of tests all distillations were carried out cracking to coke at a pressure of 150 lb. per sq. in. All experiments were carried out under exactly the same conditions, and yields are strictly comparable.

Cracking Stock.	Yield of Cracked Spirit, % by volume.
Crude shale oil	62·0
Crude shale oil distillate	63·0
Acid and soda treated crude shale oil distillate	65·7
Blue oil	65·0
Heavy oil and paraffin	68·4
Paraffin wax	78·9
Kerosene	83·0

Refining of Cracked Spirit.

Cracked shale spirit as produced by liquid-phase low-pressure cracking is more saturated than spirit recovered from the retort gases, and this probably explains the comparatively low octane rating.

	Spirit Recovered from Shale Retort Gas, %.	Cracked Spirit from Shale Oil, %.
Saturated hydrocarbons	42·5	56·0
Unsaturated hydrocarbons	48·0	32·0
Naphthenes	9·5	12·0
Aromatic hydrocarbons	Nil.	Nil.

The principal impurities to be removed are pyridine bases, phenolic bodies and gum-forming unsaturated hydrocarbons. As with straight-run spirit, the most successful refining agent known at present is sulphuric acid, but in this case to remove the bulk of the phenols (which amount to 1 per cent.), a preliminary soda treatment is necessary. The refining scheme therefore consists of caustic soda, acid, caustic soda and re-running, followed by plumbite and sulphur treatment of the distillate. Several interesting features present themselves in this operation. The acid treatment must be controlled within fairly narrow limits; a low acid treatment leaves pyridine bases in the spirit, with consequent bad smell, which is not removed by the subsequent sweetening process. On the other hand, too much acid gives a distillate of high SO_2 content, which requires excess soda to neutralize it before plumbite, and at the same time the pressure distillate residue becomes acidic, and is therefore not at all suitable for cracking stock. Excess acid treatment also results in carbon formation in the pipe-still and tower, thereby reducing the time efficiency of the unit.

To prevent the formation of SO_2, re-running of pressure distillate should be carried out at as low a temperature as possible. High temperatures favour the evolution of SO_2, which must be removed, since it adversely affects the subsequent plumbite treatment and causes severe corrosion of the distillation equipment.

The plumbite process employed is of the type where plumbite is regenerated *in situ*, and is conducted on the batch system with pump circulation. The mercaptan content of the spirit is 0·005 per cent. and average plumbite and sulphur consumption works out at about 0·125 per cent. plumbite solution (0·2 lb. PbO and 1·5 lb. NaOH per gallon) and 0·0002 lb. per gallon of sulphur. The presence of phenols complicates the operation as, should the preliminary soda wash be inefficient, the concentration of caustic soda decreases rapidly and the lead combines with phenols. In particularly bad batches this is evident by the spirit going off colour and ultimately throwing down a deposit of lead phenates, but it is possible to get an apparently water-white clear spirit which, on exposure to light and/or heat, becomes cloudy. It has been shown that by washing the spirit with water, the lead phenates, even from apparently clear and colourless spirit, are removed as a sludge, so that water-washing becomes a necessary operation to ensure a satisfactory product. Another feature of the plumbite when applied to shale cracked spirit is that the small quantity of phenols still in the spirit is extracted by the caustic soda, and the sodium phenate solution so formed at the same time is oxidized, and ultimately this coloured material redissolves in the spirit, and is not removed by washing with water. At this stage the plumbite solution must be discharged, and so a careful control of the preliminary soda wash is essential.

(c) *Hydrogenation.*

Theoretically hydrogenation is the most efficient way of refining shale oil, and actually yields of 100 per cent. by volume and over can be realized. In all other known methods of refining, these troublesome compounds, pyridine bases and phenols, peculiar to shale and coal oils, have to be removed, and incidentally the most active unsaturated hydrocarbons are lost. In hydrogenation, however, the elements, nitrogen, oxygen and sulphur, are replaced by hydrogen, and the unstable olefines and diolefines become saturated to the more stable paraffins.

Scotch shale oil can readily be hydrogenated to motor spirit. Various methods of treating the material are possible, and one method by which yields of slightly over 100 per cent. by volume can be realized is briefly outlined.

The shale oil is first distilled, leaving a residue above the diesel oil range. This residue is hydrogenated in the liquid phase whereby it is converted into 5 to 10 per cent. motor spirit and gas oil plus recycle oil. These are separated by distillation, the gas oil blended with the distillate from the topping unit and the blend subjected to hydrogen in the vapour phase. The products of this operation are spirit and re-cycle oil, which are again separated by distillation and the gas oil re-cycled. The benzine produced is water-white, and only requires a caustic-soda wash to make it marketable. The Octane No. of motor spirit so produced is low—about 51–54.

The Octane No., however, can be increased, but unfortunately only at the expense of yield. Two methods of raising the Octane No. are—hydrogenation at a higher temperature or re-forming. It has been estimated that to get an Octane No. of 72 by the former method, the yield would be reduced by 17–20 per cent. by volume. By reforming, the octane rating could probably be increased by about 10 Octane Nos. with a decrease in yield of 20 per cent.

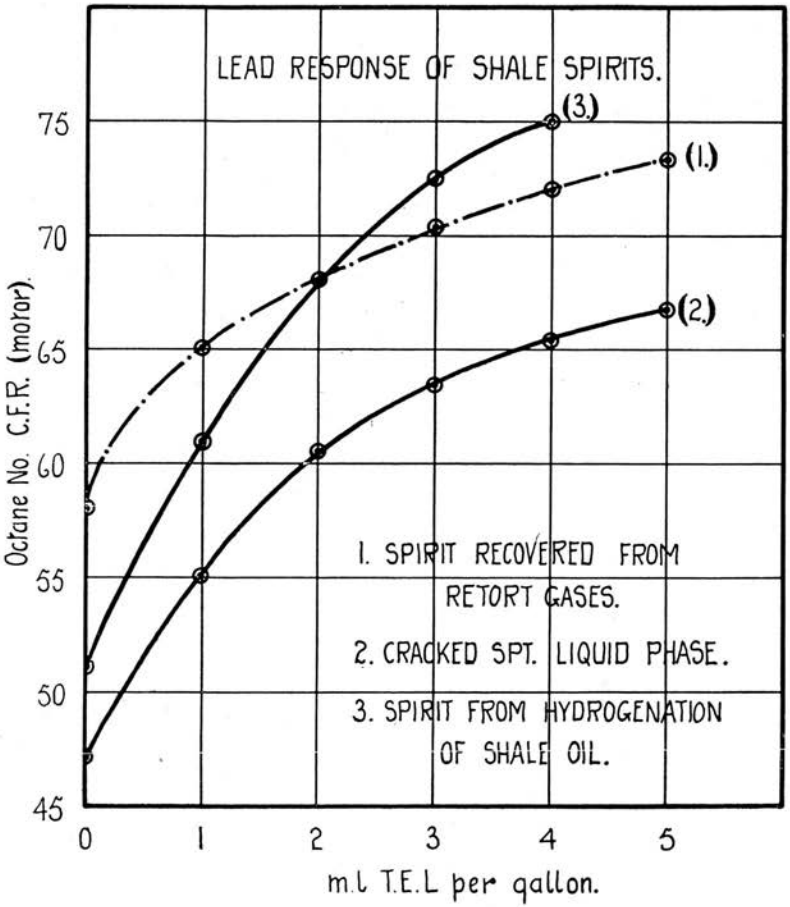


FIG. 4.

Another method of increasing the octane rating is by the addition of tetra-ethyl lead, and in this case, probably owing to the comparative freedom of the spirit from sulphur, the lead response is very good; the addition of 2·5 mls. per gallon is sufficient to raise the Octane No. from 54 to 70.

For comparison, analyses of shale straight-run spirit, shale cracked spirit, and the spirit obtained by hydrogenation of shale oil are given in Table XIII. The lead response curves of these three spirits are shown in Fig. 4.

TABLE XIII.

	Spirit Recovered from Retort Gases.	Cracked Spirit Liquid Phase.	Spirit from Hydrogenation of Shale Oil.
Sp. gr.	0·732	0·740	0·720
Iodine value (Wijs 1 hr.)	116	85	—
Bromine No.	60	40	—
Aniline point	42·0° C.	48·3° C.	60·3° C.
Sulphur	0·06%	0·10%	0·003%
I.B.P.	49° C.	36° C.	38° C.
10% dist. at	79 ,,	71 ,,	70 ,,
20% ,,	88 ,,	92½ ,,	87 ,,
30% ,,	96 ,,	111 ,,	100 ,,
40% ,,	104 ,,	126 ,,	112 ,,
50% ,,	111 ,,	139½ ,,	122 ,,
60% ,,	119 ,,	151½ ,,	134 ,,
70% ,,	127½ ,,	163 ,,	145 ,,
80% ,,	138 ,,	174 ,,	158 ,,
90% ,,	152 ,,	185 ,,	174 ,,
F.B.P.	178 ,,	200 ,,	194 ,,
Total distillate	98·0%	98·0%	98·0%
Residue	1·0%	1·0%	1·0%
Loss	1·0%	1·0%	1·0%

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DISCUSSION.

DR. K. WISSEL said :—With respect to the octane number of the petrol obtained by hydrogenation as given in this paper, I wish to mention that experiments which have been carried out using newer catalysts, have given petrol with octane number of 65–67 C.F.R., which is partly due to the formation of *iso*paraffins. These results are reported in the paper by M. Pier.

DR. A. E. DUNSTAN stressed the importance of preparing diesel oils rather than motor fuel, with the concomitant high loss of gas. It appeared to him that the most effective way of handling shale oil was *via* the diesel fuel or alternatively the production of reactive gas for synthetic purposes. He congratulated the authors on new points of importance on refining shale oils.